
**Quality Assurance Project Plan
for
McDonnell Douglas RFI
Hazelwood, Missouri Facility
Volume II, Appendix A**

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May 29, 1997

ESE Project No. 5197-042-0100



**R00058111
RCRA Records Center**

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1.0 Project Description

1.1 Introduction

As part of the McDonnell Douglas (MD) Facility's RCRA Part B Permit, MD has agreed to perform a RCRA Facility Investigation (RFI) at its Facility in Hazelwood, Missouri. This QAPP and the associated RFI Workplan present MD's approach to characterize potential releases from five solid waste management units (SWMUs) identified in the Permit.

1.2 QAPP Preparation Guidelines

This QAPP has been prepared in accordance with the Facility's Part B Permit. Please refer to Section 3 of the RFI Workplan for discussions of:

- Site/Facility Description;
- Location;
- Facility/Size and Borders;
- Topography and Surface Drainage;
- Local Geology & Hydrogeology;
- Site/Facility History;
- Past Data Collection Activities; and,
- Current Status.

This QAPP presents the policies, organization, objectives, functional activities, and specific quality assurance and quality control activities designed to achieve the data quality goals of the RFI. The QAPP shall also include the RFI objectives, sampling procedures, analytical methods, field and laboratory quality control samples, chain-of-custody procedures and data review, validation and reporting procedures.

1.3 Sample Network Design and Rationale

In order to evaluate the SWMUs, a sampling program including subsurface sampling will be performed. The purpose of the subsurface sampling is to determine chemical concentrations in soil for specific constituents of concern. This characterization will provide a clearer understanding of the nature and extent of any potential impacts to soil for each of the five SWMUs of concern at the Facility.

The soil samples will be collected from selected locations associated with the five SWMUs using the Geoprobe sampling technique. A summary of the surface and subsurface sampling is

presented below. Sampling locations are presented in Section 3.0 of the RFI Workplan, Figures 3-1 through 3-5. The specific sampling depths, number of samples, and the number of soil borings may be modified based on field observations and screening. The selection of analytical parameters is based on the results of the preliminary RFA, and RCRA Closure sampling and analysis.

- **SWMU No. 17**--Collect two samples each from three soil borings (total of six samples) with anticipated sample depths of 1-2 ft bls and 5-6 ft bls. Samples will be analyzed for metals and volatile organic compounds (VOCs).
- **SWMU No. 21**--Collect two samples each from six soil borings (total of 12 samples) with anticipated sample depths of 1-2 ft bls and 24-25 ft bls. Samples will be analyzed for metals and cyanide.
- **SWMU No. 26**--Collect two samples each from three soil borings (total of six samples) with anticipated sample depths of 1-2 ft bls and 5-6 ft bls. Samples will be analyzed for metals and VOCs.
- **SWMU No. 31**--Collect two samples each from three soil borings (total of six samples) with anticipated sample depths of 1-2 ft bls and 5-6 ft bls. Samples will be analyzed for metals, polynuclear aromatic hydrocarbons (PAHs) and VOCs.
- **SWMU No. 10**--Collect two samples each from three soil borings (total of six samples) with anticipated sample depths of 1-2 ft bls and 5-6 ft bls. Samples will be analyzed for metals, PAHs, and VOCs.

1.3.1 Field Parameters

Soil samples will be screened in the field for organic vapors, metals, and waste oil constituents.

1.3.2 Analytical Parameters

The projected analytical parameters and their associated detection limits are presented in Table 1-1.

1.3.3 Data Quality Levels

The laboratory detection levels for VOCs, PAHs, RCRA metals, and cyanide are presented in Table 1-1. These detection levels will meet the project objectives.

2.0 Project Organization and Responsibility

This section describes the structural organization and assigned responsibilities for the QA portion of the RFI. MD retains overall responsibility to perform and maintain the RFI activities presented in the RFI Workplan and this QAPP for the Facility. Please refer to Section 2.7 of the RFI Workplan for details regarding the overall project organization and responsibilities.

ESE Laboratories in Peoria, Illinois will perform the required laboratory analyses and data validation tasks in accordance with this QAPP. Additional detail regarding laboratory-specific lines of authority, reporting, and responsibilities are described below.

2.1 Management Responsibilities

MD Project Manager

The MD Project Manager is Joe Haake. The MD Project Manager will be involved with the implementation and maintenance of RFI activities. His quality assurance related responsibilities will include the following:

- Define RFI objectives and develop a detailed work plan schedule;
- Establish project policy and procedures to address the specific needs of the RFI as a whole;
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements and authorizations;
- Approve all reports (deliverables) before their submission to MDNR;
- Ultimately be responsible for the preparation and quality of all reports; and,
- Represent the project team at meetings.

ESE Project Manager

The ESE Project Manager is Doug Marian. The ESE Project Manager has responsibility for ensuring that the project meets the RFI objectives and quality standards as established in this QAPP, as well as the associated RFI Workplan. The ESE Project Manager will report directly to the MD Project Manager.

2.2 Quality Assurance Responsibilities

ESE QA Manager

The ESE QA Manager is Lana Smith. The ESE QA Manager reports directly to the ESE Project Manager and also has a line of communication to the MD Project Manager. The ESE QA Manager will be responsible for ensuring that all RFI procedures for this project are being followed.

Additional specific functions and duties include:

- Reviewing and approving QA plans and procedures;
- Providing QA technical assistance to project staff;
- Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the ESE Project Manager; and
- The ESE QA manager is responsible for review of field and analytical data generated by the field team to ensure it meets the RFI requirements.

2.3 Laboratory Responsibilities

ESE Laboratories Project Manager

The ESE Laboratories Project Manager is Vickie Wynkoop. The ESE Laboratories Project Manager will report directly to the ESE QA Manager and also maintain communication with the ESE Laboratory Data Validator and will be responsible for the following:

- Ensuring all laboratory resources are available on an as-required basis; and,
- Reviewing all final analytical reports.

ESE Laboratories Operations Manager

The ESE Laboratories Operations Manager will be responsible for:

- Coordinating laboratory analyses;
- Supervising in-house chain-of-custody;
- Scheduling sample analyses;
- Overseeing data review;
- Overseeing preparation of analytical reports;
- Recommending corrective actions, if needed, to the ESE QA Manager; and,
- Approving final analytical reports prior to submission to MD.

ESE Laboratories QA Manager

The ESE Laboratories QA Manager has the overall responsibility for data after it leaves the laboratory. The ESE QA Manager will be independent of the laboratory but will communicate data issues through the ESE Project Manager. In addition, the ESE QA Manager will:

- Implementing specified QC procedures for data acquired provided by the technical field staff;
- Authoring and writing reports of Field Team activities;
- Identifying problems at the Field Team level, resolving difficulties in consultation with the MD and ESE Project Managers, implementing and documenting corrective action procedures, and provision of communication between the Field Team and other project team managers; and,
- Participating in preparation of the RFI Report.

ESE Field Technical Staff

The ESE technical field staff for this project will be drawn from ESE's local pool of professional/technical staff. The ESE technical field team staff will be utilized to gather and analyze data, and to prepare various task reports/support materials. All of the designated ESE technical field team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

3.2.3 Analysis Accuracy Objectives

Analysis accuracy is assessed through the evaluation of matrix spikes and matrix spike duplicates (MS/MSD), matrix duplicates, Laboratory Control Samples (LCS) and the determination of percent recoveries. Results of the LCS in conjunction with the MS/MSD can be used to provide evidence the laboratory performed the method correctly and, if applicable, the extent of matrix interference.

3.3 Completeness

3.3.1 Definition

Field and laboratory completeness is the number of valid measurements obtained from all measurements planned to be taken in the field or laboratory, respectively.

3.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all measurements planned to be taken in the field. The equation for completeness is presented in Section 12.0 of this QAPP. For the RFI, field measurements will consist of organic vapor headspace, UV/fluorescence, and XRF screening methods. Field completeness for organic vapor and XRF screening measurements will be 80 percent. Field completeness will not apply to the UV/fluorescence screening activities, as these efforts are for qualitative screening purposes.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all measurements planned to be taken in the laboratory. The equation for completeness is presented in Section 12.0 of this QAPP.

Laboratory completeness will be 80 percent.

3.4 Representativeness

3.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter, variations at a sampling point, a process condition, or an environmental condition.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the sampling procedures presented in Section 4.0 of the RFI Workplan are followed and that proper sampling techniques are used.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using proper analytical procedures for the appropriate target analyte, sample matrix, detection limit and method. The sampling network was designed to provide data necessary to characterize potential releases to soil. During development of this network, consideration was given to the operational history of the facility, past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the RCRA program.

3.5 Comparability

3.5.1 Definition

Comparability is an expression of the confidence with which one data set can be compared with another.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the procedures referenced in Section 4.0 of the RFI Workplan are followed and that proper sampling techniques are used.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used as documented in this QAPP. Comparability is also dependent on similar QA objectives.

3.6 Level of Quality Control Effort

Method blank, field duplicate, and MS/MSD samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples.

One field duplicate sample will be collected at a rate of 1 duplicate per 20 analytical samples. Based on the currently anticipated scope of work, two field duplicates will be collected for this project. Similarly, one MS/MSD sample will be analyzed for every 20 or fewer investigative samples. For "solid" samples, additional sample volume is not required.

4.0 Sampling Procedures

Sampling procedures to be utilized at each of the five SWMUs will be consistent with the objectives of the investigation. Sampling procedures are described in Section 4.0 of the RFI Workplan which is being submitted with this QAPP and is incorporated herein by reference. Please refer to the RFI Workplan for sampling protocols.

5.0 Custody Procedures

Custody is one of several factors that are necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, will be maintained under document control in secure areas.

A sample or evidence file is under your custody if:

- the item is in actual possession of a person;
- the item is in the view of the person after being in actual possession of the person;
- the item was in actual physical possession but is locked up to prevent tampering; or,
- the item is in a designated and identified secure area.

5.1 Field Custody Procedures

Field data collection activities will be recorded using field logbooks. As such, entries will be described in as much detail as possible so that on-site field team members can reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- person to whom the logbook is assigned;
- logbook number;
- project name;
- project task start date; and,
- project task end date.

Logbook entries will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all field team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the investigation area and the purpose of their visit will also be recorded in the field logbook.

Descriptions of any measurements or collected samples will be recorded. All entries will be made in ink, signed or initialed and dated, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark which is signed or initialed and dated by the sampler. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station shall be recorded. The number of the photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Notes will also be recorded to document other sampling specifics including equipment used, time of sampling, sample description, depth of sample collection, number of sample containers, and container volume. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive a separate sample identification number, will be noted under sample description.

The sample packaging and shipment procedures will ensure that the samples will arrive at the laboratory with the chain-of-custody intact. An example chain-of-custody form is provided in the Laboratory QAPP (Appendix A).

- a. The field sampler will be personally responsible for the care and custody of the samples until they are transferred or properly dispatched.
- b. All bottles will be identified by use of sample labels with sample numbers, sampling locations, and the date/time of collection.
- c. Sample labels will be completed using waterproof ink unless prohibited by weather conditions.
- d. Samples will be accompanied by a properly completed chain-of-custody form which contains the associated sample numbers and locations. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents the custody transfer of samples from the sampler to another person, to the permanent laboratory, or to/from a secure storage area.
- e. Sample containers will be wrapped individually in "bubble pack" and placed on ice at 4°C in a sample box or cooler. Insulation material such as styrofoam peanuts or additional bubble pack will be used to fill any remaining void space in each sample box or cooler. Samples will be shipped to the ESE laboratory with a signed chain-of-custody record secured to the inside top of each shipping container. Shipping containers will be

secured with strapping tape and custody seals for shipment to the laboratory. Custody seals will be attached to the cooler. The custody seals will be signed by the Field Implementation Manager before they are attached to the shipping container.

5.2 Laboratory Custody Procedures

Samples are received by the ESE Sample Custodian who records and files all shipping documentation. The Sample Custodian has full responsibility for ensuring that proper custody procedures are followed at the laboratory and that project specific files are maintained. Upon receipt by ESE, samples proceed through an orderly processing sequence designed to measure continuous integrity of both the sample and its documentation. Upon receipt of a sample shipment, the Sample Custodian initiates a Sample Log-in Checklist for each sample shipment. Custody seals on coolers remain intact until the Sample Custodian is ready to log-in the specific set of samples contained in the cooler. Coolers are inspected for proper seals and to ensure the seals are intact.

The cooler is opened and the internal temperature of the cooler is taken on a temperature blank contained within the cooler. Lacking a temperature blank, the temperature of a representative sample is measured using an infrared thermometer. The samples are then unpacked, inspected and checked against the accompanying chain-of-custody record. Any discrepancies involving sample integrity, sample breakage, cooler temperature, appropriate container use, preservatives, and missing or incorrect documentation are immediately noted on the Sample Log-in Checklist. If inconsistencies, discrepancies or inadequacies with respect to the received samples are identified, the Sample Custodian will notify the ESE Project Manager and Operations Manager who is responsible for resolving the problem. Resolution typically will involve contacting the field sampling team with follow-up documentation of conversations and resolution. Samples will not be logged until the problems are resolved. (See Section 13.3 of this QAPP for discussion on Laboratory Corrective Action).

Once all sample shipment problems have been resolved (if any), the Sample Custodian will log the samples into ESE's tracking log and transfer the sample information to the laboratory's electronic database.

A unique laboratory identification (ID) number will be assigned to each sample at the time of logging. Sample numbers will be assigned sequentially. Sample numbers will be used on all paperwork associated with the sample so that all documentation throughout the laboratory can be matched to the appropriate sample.

The samples are logged into the laboratory's electronic database. The information recorded in the database includes the field identification number, the laboratory identification number, date and time of receipt in the laboratory, and date and time of sample collection. Additional pertinent comments may also be recorded. The initials of all personnel who handled the samples are also manually written on the hard copy of the log-in paperwork.

Samples are assigned a storage location during the log-in procedure. Assignment is made based on the storage requirements for each sample and test method. Samples are stored in one of two locations: a walk-in refrigerator and a VOA refrigerator. The VOA refrigerator is located in the laboratory facility; access is controlled by limiting access to the facility. Each sample will remain in its storage location until the time of analysis. The samples are removed by the analysts and returned as soon as possible.

No chemical standards are kept in the walk-in or VOA refrigerators. Instead, they are segregated from the samples and are kept in the laboratory where they are used.

All samples and sample extracts will be retained after analysis is complete. Unused portions of samples and sample extracts will be disposed of 30 days after the delivery of final report delivery unless otherwise specified.

A case file will be created for the program. Project information including the final report, invoice, client contact notes, chain-of-custody, and all relevant paperwork are contained in the case files. After project completion, an inventory of the case files will be created and transferred along with the contents of the case files to a storage box.

5.3 Final Evidence Files

The final evidence file (FEF) will consist of all documents relevant to the sampling and analysis activities described in this QAPP which includes, reports, logs, field notebooks, pictures, subcontractors reports, and data reviews that relate to the sampling and analysis activities.

The final evidence file will include at a minimum:

- field logbooks;
- field data and data deliverables;
- drawings;
- laboratory data deliverables;
- data validation reports;
- data assessment reports;

- progress reports, QA reports, interim project reports, etc.;
- all custody documentation (forms, airbills, etc.); and
- laboratory project folders and storage boxes.

6.0 Calibration Procedures and Frequency

This section describes the calibration procedures and the frequency at which these procedures will be performed for both field and laboratory instruments.

6.1 Field Instrument Calibration

As part of the RFI, organic vapor headspace, UV/fluorescence, and XRF screening activities will be performed in the field on soil samples. As a general rule, the organic vapor detector and XRF instruments will be calibrated prior to use each day. The UV/fluorescence instrument does not require calibration since it only provides a qualitative reading.

Calibration procedures will be documented in the field logbook and will include the date/time of calibration, name of person performing the calibration, reference standards used, and the readings. Multiple readings on one sample or standard, as well as readings on replicate samples, will likewise be documented.

Organic Vapor Detector Calibration

The organic vapor detector will be a photoionization detector (PID). The PID will be calibrated to report the response in parts per million relative to the potentiometric response of isobutylene. The PID will be calibrated using a certified gas standard containing 100 ppm (accurate to within 2 percent) of isobutylene in air. The calibration procedure is described below.

- Connect the cylinder of calibration gas to the probe tip of the PID.
- Set the flow rate of calibration gas into the PID at 0.25 liters per minute.
- Adjust the PID meter response to read 100 ppm by manually adjusting the "span" setting on the instrument.
- Record the span setting in the calibration log book that is kept with each instrument.

X-Ray Fluorescence Meter Calibration

The x-ray fluorescence (XRF) meter to be utilized will be a Spectrace Instruments model Spectrace 9000 FPXRF analyzer. This XRF unit is supplied with three factory-installed XRF

calibrations. One of these internal calibrations is specifically designed for soil screening applications. This internal calibration feature will be verified by screening a known standard on a daily basis prior to use.

6.2 Laboratory Instrument Calibration

The ESE laboratory maintains a variety of logbooks documenting calibration procedures and results. Logs of balance calibrations, chemical receipt, and standard preparation are maintained by the sample preparations facility. A log of instrument calibration and usage is maintained by each instrumental facility.

Calibration of laboratory equipment will be based on approved written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing quality control activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will maintain competent repair staff with in-house spare parts or will maintain service contracts with vendors.

6.2.1 Gas Chromatograph/Mass Spectrometer (GC/MS)

Tuning

For the analysis of volatile organic compounds by full scan GC/MS, the detector is tuned using 4-bromofluorobenzene (BFB). A check of the tuning is made at the beginning of each analytical sequence and every twelve hours of instrument operation.

The tuning checks must meet criteria before any standards or samples may be analyzed. Standards and samples must be analyzed under the same settings as those used to check detector tuning.

Initial Calibration

The linear range of each method is determined by the analysis of calibration standards at five levels. To demonstrate acceptable minimum response, the relative response factor (RRF) for each compound in each calibration level is calculated. To demonstrate linearity across the calibration range, the standard deviation of the RRFs for each compound expressed as a percentage of the mean RRF (percent relative standard deviation -- %RSD) is calculated.

All sample calculations are performed using the average RRF from a valid initial calibration.

Continuing Calibration

Each method is routinely checked by analyzing a continuing calibration standard to ensure that the instrument continues to meet sensitivity and linearity requirements. To demonstrate acceptable minimum response, the relative response factor (RRF) for each compound is calculated. To demonstrate the validity of the initial calibration, the RRF calculated from the continuing calibration standard and the average RRF of the initial calibration curve is compared. The difference between the values is expressed as a percentage of the initial calibration RRF (%D).

Other Laboratory Instruments

Analytical Balances

Each analytical balance is checked prior to its use to ensure its accuracy. The check is performed prior to its use and on each day that the balance is used. The check is made using Class S weights. Measurements are recorded in a log maintained by the laboratory.

Top-loading Balances

Each analytical balance is checked prior to its use to ensure its accuracy. The check is performed prior to its use and on each day that the balance is used. The check is made using Class S weight. Measurements are recorded in a log maintained by the laboratory.

Thermometers

All thermometers used are calibrated against a NIST-certified thermometer. Record of thermometer calibrations are maintained by the laboratory.

7.0 Analytical and Measurement Procedures

This section summarizes the analytical and measurement procedures that will be utilized to evaluate the soil samples collected as part of the RFI.

7.1 Field Analytical and Measurement Procedures

Quality assurance objectives for measurement of field data in terms of precision, accuracy, completeness, representativeness, and comparability are presented in Section 3.0 of this QAPP. Calibration procedures and frequency for field instruments are presented in Section 6.1 of this QAPP. Field sampling procedures are discussed in Section 4.0 of the RFI Workplan.

7.2 Laboratory Analytical and Measurement Procedures

Laboratory analyses will be performed in accordance with this QAPP. Facility-specific analytical fractions and their associated methods for analysis are provided below:

- VOCs by USEPA Method 8240;
- PAHs by USEPA Method 8310;
- Barium, cadmium, chromium, lead, and silver by USEPA Method 6010;
- Arsenic by USEPA Method 7060;
- Mercury by USEPA Method 7471; and
- Selenium by USEPA Method 7740.

8.0 Internal Quality Control Checks

8.1 Field Quality Control Checks

QC procedures for organic vapor headspace and XRF screening measurements on soil samples will include calibrating the instruments as described in Section 6.1 of this QAPP. Duplicate field measurements will be taken as stated in Section 3.1.2 of this QAPP (e.g. 1 duplicate per twenty samples). Assessment of field sampling precision and bias will be made by collecting field duplicates of soil samples for laboratory analysis. Collection of the samples will be in accordance with the applicable procedures in the RFI Workplan.

8.2 Laboratory Quality Control Checks

The following quality control measures and checks will be employed by ESE for the organics fraction of this program:

- Method and procedural blanks to assess the level of contamination associated with the processing and analysis of samples;
- Blank Spike (BS) samples consisting of representative target analytes spiked into a blank matrix to assess method performance independent of sample matrix;
- Matrix spikes and matrix spike duplicates (MS/MSD) samples to assess method performance in the subject matrix;
- Surrogate compounds to monitor the efficiency of the analytical procedures; and
- Analysis of samples within generally accepted method holding times.

8.3 Specific Quality Control Assignments by Sample Group

Definition of Batches

The following definitions are used:

- Sample Delivery Group or QC Batch--A group of samples received together (or over a few days) with a specific QC assignment. Applied to all samples.
- Preparation Batch or Extraction Batch--A group of 20 or fewer field samples plus associated QC samples prepared together. Usually applied to semivolatile organics analysis.
- Instrument Batch or Analytical Sequence--A group of individual instrumental analyses sequenced in a prescribed order.

Project QC

Specific laboratory QC samples will be analyzed as follows:

<u>Element</u>	<u>Volatile Organics Analysis</u>	<u>Semivolatile Organics Analysis</u>
Procedural Blank	One per instrument batch	One per preparation batch
Blank Spike	One set per twenty field samples analyzed	One per extraction batch
Matrix Spike and Matrix Spike Duplicate	As assigned (one set per twenty field samples)	As assigned (one set per twenty field samples)

All data obtained will be properly recorded. It is expected that sufficient volumes of samples will be collected to allow for reanalysis when necessary.

8.4 Quality Assurance Objectives

Quality assurance objectives can be expressed in terms of precision, accuracy, representativeness, comparability, and completeness. Section 12.0 of this QAPP lists QA objectives for measurement data in terms of precision, accuracy, and completeness. Adherence to the data quality objectives will be quantitatively measured by comparing the results of field and QC sample analyses to prescribed control limits as detailed below.

8.5 Control Limits

Control limits are created for all QC parameters. These limits may be based on historical results or set considering the accuracy and precision requirements of the resultant analyses.

8.6 Holding Times

Sample analysis will be scheduled to meet all method holding times. A best effort will be made to complete extraction and analysis before the holding time for preparation has expired so that samples can be re-extracted within holding time should problems arise.

Every attempt will be made to meet holding time for the preparation of re-extracted samples. If samples are being re-extracted outside of holding time, the ESE Laboratory Project Manager will immediately notify the ESE and/or MD Project Managers. Any and all nonconformance situations will be fully documented in the report narrative.

8.7 Blank Spike Samples

One blank spike sample is prepared with each batch of 20 or fewer field samples. Where one or more of the spiked analytes does not meet the accuracy criteria, all associated samples are re-prepared and re-analyzed unless evidence is present that supports accepting all data.

8.8 Matrix Spike and Matrix Spike Duplicate Samples

One set of matrix spike/matrix spike duplicates (MS/MSD) is prepared and analyzed with each batch of 20 or fewer investigative samples. Recovery and relative percent difference for the spiked compounds is calculated and compared to acceptance limits. The laboratory will use the following to evaluate the QC results:

1. For samples with results within "Acceptance Limits," data will be accepted and reported.
2. For samples with results outside "Acceptance Limits" but within "Warning Limits," results of the associated laboratory QC results (blank, blank spike, surrogate recoveries) will be evaluated. If laboratory QC results are within limits, the sample results will be accepted and reported.
3. Samples with results outside "Warning Limits" will be re-extracted and re-analyzed. If the reanalysis supports the initial analysis, the initial analysis will be reported with a discussion of the corrective action in the project narrative. If the reanalysis yields results within limits, the reanalysis will be reported.

Although not expected, there may be other situations where re-extraction and re-analysis may not be required:

- MS/MSD samples require significant dilution due to the concentrations of target compounds present beyond the linear range of the instrument. In this case, the matrix spike compounds may be so dilute as to be unmeasurable. An attempt to compensate for this will be made at the time of sample preparation.
- Target analytes in the MS/MSD sample are at levels significantly higher than that spiked. Again, an attempt will be made to compensate for this at the time of sample preparation.
- The sample is characterized by significant chromatographic interference. This is minimized by the use of sample cleanups and selected ion monitoring. Additional cleanups will be considered if this occurs.

8.9 Surrogate Compounds

Surrogates are spiked into all field and QC samples for organic analyses. Recovery of the spiked compounds is calculated and compared to acceptance limits. The laboratory will use the following to evaluate the QC results:

1. For samples with results within "Acceptance Limits," data will be accepted and reported.
2. For samples with results outside "Acceptance Limits" but within "Warning Limits," laboratory QC results (blank, blank spike, surrogate recoveries) will be evaluated. If laboratory QC results are within limits, the sample results will be accepted and reported.
3. Samples with results outside "Warning Limits" will be re-extracted and re-analyzed. If the reanalysis supports the initial analysis, the initial analysis will be reported with a discussion of the corrective action in the project narrative. If the reanalysis yields results within limits, the reanalysis will be reported.

Although not expected, there may be other situations where re-extraction and re-analysis may not be required:

- The sample requires significant dilution due to the concentrations of target compounds present beyond the linear range of the instrument. In this case, the surrogate compounds may be so dilute as to be unmeasurable. An attempt to compensate for this will be made at the time of sample preparation.
- The sample is characterized by significant chromatographic interference. This is minimized by the use of sample cleanups and selected ion monitoring. Additional cleanups will be considered if this occurs.

8.10 Procedural Blanks

For volatile organics by GC/MS, the concentration of each target compound found in the blank must be less than the minimum reporting limit except for methylene chloride, acetone, and 2-butanone, which must be less than 5 times the minimum reporting limit.

For semivolatile organics by HPLC, one procedural blank will be prepared and analyzed with each batch of 20 or fewer field samples. No target compound may exceed the minimum reporting limit. If one or more of the target analytes is detected above the minimum reporting limit, laboratory contamination is suspected and the associated samples are re-prepared and re-analyzed.

9.0 Data Reduction, Validation, and Reporting

Data generated through RFI field sampling activities or by the laboratory operation shall be reduced and validated prior to reporting. No data shall be disseminated until it has been subjected to the procedures which are summarized in subsections below.

9.1 Data Reduction

9.1.1 Field Data Reduction Procedures

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Only direct-read instrumentation will be employed in the field. The field instruments will generate measurements directly read from the meters following calibration per manufacturer's recommendations as outlined in Section 6.1 of this QAPP. Such data will be written into field log books immediately after measurements are taken. If errors are made, results will be legibly crossed out, signed or initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, when the results tables and figures required for this study are being completed, the Field Implementation Manager will proof the tables and figures to determine whether any transcription errors have been made by the technical field staff.

9.1.2 Laboratory Data Reduction Procedures

This section presents ESE's Laboratory Data Reduction Procedures. ESE will perform data reduction and internal validation under the direction of the ESE QA Manager. The ESE QA Manager is responsible for assessing data quality and advising of any data which were rated "preliminary" or "unacceptable" or other notations which would caution the data user of possible unreliability.

All analytical data generated are extensively checked for accuracy and completeness. The data validation process consists of data generation, data reduction, and three levels of review, as described below.

After acquisition, the raw data is reduced into reportable values by the analyst using computer software. Additional sample information is added to the sample results during data reduction by the analyst. Identification of target analytes is first performed by the computer software and then checked by the analyst. Each chromatographic integration is also checked. Missed target analytes and misidentified analytes are corrected by the analyst. The finished results are then converted electronically for use in the data reporting software.

The analyst is responsible for reviewing the sample and QC results for compliance to this QAPP. QC exceptions are immediately brought to the attention of the ESE Laboratory Project Manager or the ESE Laboratory QA Manager. Corrective action for problems are made where necessary.

The analyst then assembles hard copies of the computer software output into a final laboratory data package. Additional relevant supporting documentation, including sample and standard preparation record are also added to the final laboratory data package. The completed package is submitted to the facility supervisor for review.

The audit process is coordinated by the ESE Laboratory QA Manager. The formal audit process includes a 100% review of all hand calculated values and a 10% review of computer generated results. The process checks the traceability of a final result through the instrument calibration and to the sample preparation steps. A formal report is issued to the responsible analysts and facility supervisors at the completion of the audit for response. Upon completion of the responses, the auditor will release the results to the ESE Laboratory Project Manager for review and reporting. The final data package and the audit report are maintained in the laboratory files. The ESE Laboratory Project Manager is responsible for completing the project narrative letter and assembling the package for final reporting.

9.2 Data Validation

Data validation procedures shall be performed for both field and laboratory operations as described below.

9.2.1 Field Data Evaluation and Validation Procedures

After completing a sampling program, the field data package (field logs, calibration records, chain-of-custody forms, etc.) will be reviewed by the ESE Project Manager or their representative for completeness and accuracy. Items to be considered in the Field Data Package Validation Procedures will include but are not limited to the following:

- a. A review of field data contained on field sampling logs for completeness.
- b. A verification that field replicates were properly prepared, identified, and analyzed.
- c. A check on field analyses for equipment calibration and condition.
- d. A review of chain-of-custody forms for proper completion, signatures of field personnel and the laboratory sample custodian, and dates.

The field data packages will undergo 10 percent data validation review.

If a problem is identified the percentage level of data validation will increase until the problem is identified and solved. Once the problem is solved the percentage level of data validation will decrease back to the 10 percent level.

Field data package validation review will be performed by the ESE QA Manager.

9.2.2 Independent Laboratory Data Validation

Validation of laboratory data will be performed by the ESE Laboratory Data Validator upon receipt of the laboratory data packages.

Ten percent of the laboratory data will be validated back to the raw data.

If a problem is identified the percentage level of laboratory data validation will increase until the problem is identified and solved. Once the problem is solved the percentage level of laboratory data validation will decrease back to the 10 percent level.

The data validators will utilize the appropriate and applicable USEPA guidelines such as the "National Functional Guidelines for Organic and Inorganic Data Review" (with applicable revision for SW 846 methods), the appropriate QA objectives, the results of the data evaluation, and professional judgement to make any decisions regarding interpretation of the data or impact of quality problems on the results. The guidelines are particularly useful for their standardized approaches to evaluating blank contamination, matrix interferences, instrument calibration problems, and other analytical controls impacting data quality. The actual quality control "windows" and criteria will be obtained from the methods used and Project QA requirements.

Items to be considered in the data package validation procedure will include, but are not limited to, the following:

- a. A comparison of sampling dates, sample extraction dates, and analysis dates to check that samples were extracted and/or analyzed within proper holding times.
- b. A review of analytical methods and required detection limits to verify that they agree with the project QAPP and the laboratory contract.
- c. A review of laboratory blanks to evaluate possible contamination sources; consideration should be given to preparation techniques and frequencies, as well as the analytical results.
- d. A review of field replicate data for evaluation of sampling and analytical precision.

- e. A review of laboratory QA data (tuning and calibration checks, blanks, matrix spike recoveries, matrix spike duplicate recoveries and RPD, surrogate spike recoveries, laboratory control sample recoveries, QC check sample recoveries, laboratory duplicate recoveries and RPD's linearity checks, etc.) for compliance with required acceptance criteria.

The final step in the actual validation process is interpreting and evaluating the raw data. Mass spectral interpretation is an important part of evaluating organic GC/MS analyses. Because much of the actual compound identifications are compiled by computer library matching schemes, the compound "hits" will be examined by an experienced validator to confirm that the compound identifications are correct. Quantitations of reported compounds must also be verified to assure that the quantitations are based on the correct nearest internal standard (or other appropriate criteria).

9.3 Data Reporting

Data reporting procedures shall be carried out for field and laboratory operations as indicated below.

9.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of tables and/or figures containing tabulated results of all measurements made in the field, and documentation of all field calibration activities.

9.3.2 Laboratory Data Validation Reporting

A data validation report will be prepared for every sample delivery group received. The data validation report will be based on the results of the data validation process. As a minimum, every data validation report will contain the following information:

- a. Laboratory name
- b. Site name
- c. Sample number
- d. Sample results
- e. Data Qualifiers
- f. Overall data assessment
- g. Explanation of action taken
- h. Comments

The data quality flags are identical to the system employed by the EPA for assessing CLP and similar data. The data quality flags are:

- R Code: Data flagged with an "R" has not met the required analytical QA requirements. This data is unusable even if field QC is acceptable.
- J Code: Data flagged with a "J" has not met some of the analytical QA requirements; however, the problem was not of sufficient magnitude to warrant classifying the data as unusable. Data in this category is qualitative (estimated) provided the field data meets all criteria and the sample is valid.
- U Code: The material was analyzed for, but was not detected. The associated numerical value is the sample quantification limit.
- UJ Code: The material was analyzed for, but was not detected. The sample quantification limit is an estimated value.

9.3.3 Laboratory Data Reporting

The ESE Laboratory Project Manager is responsible for the generation of the final laboratory reports. The ESE Laboratory Project Manager reviews the report to determine whether the report meets project requirements. The ESE Laboratory Project Manager will sign all reports prior to their release.

All analyses will be thoroughly documented. This documentation will be sufficient to recreate the analysis on paper. The report will consist of the tabulated results and a summary of quality control samples.

9.4 Project Files

Project files for this project will contain the following documents: correspondence between MD and ESE, chain-of-custody records, data, and a copy of the final report.

10.0 Performance and System Audits

10.1 Performance and System Audits and Frequency

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in this QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits.

10.2 Field Performance and System Audits

10.2.1 Internal Field Audits

Due to the 1-2 day duration of the RFI field activities, internal field audits are not anticipated.

10.2.2 External Field Audits

10.2.2.1 External Field Audit Responsibilities

External field audits may be conducted by the MDNR RFI Project Coordinator.

10.2.2.2 External Field Audit Frequency

External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the MDNR.

10.2.2.3 Overview of the External Field Audit Process

External field audits will be conducted according to the field activity information presented in the QAPP.

10.3 Laboratory Performance and Systems Audits

10.3.1 Internal Laboratory Audits

This section presents a description of ESE's Internal Laboratory Audits.

10.3.1.1 Internal Laboratory Audit Responsibilities

The internal laboratory audits are administered by the ESE Laboratory QA Manager.

10.3.1.2 Internal Laboratory Audit Frequency

An annual internal systems audit is conducted at the ESE laboratory by the ESE QA Manager and quality assurance staff. Internal performance audits are conducted on a semi-annual basis and are administered by the ESE QA Manager.

10.3.1.3 Internal Laboratory Audit Procedures

The internal laboratory system audits include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. The laboratory audit procedure includes an examination of the sample log-in checklists for accuracy and completeness.

The internal audits are intended to ensure that the laboratory is complying with the procedures defined in laboratory SOPs, QAPPs, and contracts. It is also designed to determine whether sample flow or analytical problems exist. The frequency of the audits will be increased if any problems are suspected.

The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The ESE QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

10.3.2 External Laboratory Audits

10.3.2.1 External Laboratory Audit Responsibilities

An external audit may be conducted at the discretion of MDNR.

10.3.2.2 External Laboratory Audit Frequency

An external laboratory audit may be conducted at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are at the discretion of the MDNR.

10.3.2.3 Overview of the External Laboratory Audit Process

External laboratory audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

11.0 Preventative Maintenance

11.1 Field Instrument Preventative Maintenance

PID, UV/fluorescence, and XRF field instrumentation used to collect data during sampling. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use with continuing calibrations being performed in accordance with calibration frequencies outlined in Section 6.1 of this QAPP. Calibration checks will be documented in the field project notebooks. Back-up instruments and equipment will be available locally or within 1-day shipment to avoid delays in the field schedule.

11.2 Laboratory Instrument Preventative Maintenance

To minimize downtime and interruption of analytical work, preventative maintenance is routinely performed on each analytical instrument. Routine maintenance includes the regular cleaning of GC inlet and column components and cleaning of the detector. Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation.

All major instrumentation are covered under service contract with external vendors. When repairs are necessary as indicated by major malfunction or inability to meet performance criteria, they are initiated by laboratory staff with consultation with the service contractor. Major service contracts include 24-hour response from the service contractor. If necessary, the service contractor is called on-site to complete the repair.

Specific routine maintenance procedures are included in the individual laboratory SOPs. Maintenance activities are recorded in logs assigned for each item of laboratory equipment.

12.0 Specific Routine Procedures Used to Assess Data Precision, Accuracy and Completeness

12.1 Accuracy Assessment

In order to assure the accuracy of the analytical procedures, an environmental sample is randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte or analytes to be evaluated. In general, a sample spike should be included in every set of 20 samples tested on each instrument. The spike sample is then analyzed. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Daily control charts are plotted for each commonly analyzed compound and kept on instrument-specific, matrix - specific, and analyte - specific bases. The percent recovery for a spiked sample is calculated according to the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \times 100$$

12.2 Precision Assessment

Aqueous samples to be spiked will be designated in the field. Soil/sediment samples to be spiked will be designated in the laboratory. The request to perform an aqueous MS/MSD will appear on the Chain of Custody form. The duplicate samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The relative percent difference (RPD) between the spike and duplicate spike are calculated and plotted. The RPD is calculated according to the following formula:

$$RPD = \frac{|\text{Amount in Spike 1} - \text{Amount in Spike 2}|}{0.5 (\text{Amount in Spike 1} + \text{Amount in Spike 2})} \times 100$$

12.3 Completeness Assessment

Completeness is the number of valid data obtained from all measurements planned to be taken in the field and laboratory. Percent completion will be calculated using the following equation:

$$\% \text{ Completeness} = \frac{V}{n} \times 100$$

where V = number of measurements judged valid

n = total number of measurements planned

13.0 Corrective Action

13.1 Corrective Action

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures (e.g. those that do not conform to the procedures set forth in this QAPP which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, or data assessment. All corrective action proposed and implemented will be documented. Corrective action will only be implemented after approval by the MD Project Manager or their designee. If immediate corrective action is required, approvals secured by telephone from the MD Project Manager will be documented in an additional memorandum.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the MD Project Manager. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPPs will be identified and corrected in accordance with the respective QAPPs.

13.2 Field Corrective Action

Corrective action in the field may be needed when the sample network is changed (i.e. more/less samples, sampling locations other than those specified in the QAPP etc.), sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. In general, the ESE Field Implementation Manager (FIM), ESE Project Manager, or the MD Project Manager may identify the need for corrective action. The ESE FIM will recommend a corrective action. The ESE FIM will bear the responsibility to ensure that the corrective action has been implemented.

If the corrective action will supplement the existing sampling plan (i.e. collection of additional samples or data) using existing and approved procedures in the QAPPs, corrective action approved by the ESE FIM will be documented. If corrective actions resulting in less samples (or analytical fractions), etc. which may cause project quality assurance objectives not to be achieved, it will be necessary that all levels of project management including the MD Project concur with the proposed action.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by the MDNR RFI Project Coordinator.

13.3 Laboratory Corrective Action

The system for reporting, evaluating, and resolving nonconformance with established quality standards is a significant component of any quality assurance plan. Need for corrective action is triggered by an identified or potential deficiency in an activity, data set, or document that may adversely affect program objectives. Corrective actions, either short-term or long-term, are instituted to eliminate the cause of nonconformance.

Corrective action needs are identified on a continuing basis through vigilance on the part of the entire laboratory staff, and on a periodic basis through a system of QA audits and reviews. If adequate corrective actions cannot be developed on an informal basis, the staff member who becomes aware of the problem is expected to notify the ESE QA Manager in writing.

Short-Term Corrective Action

With regard to data quality actions, short-term corrective actions might include, but not necessarily be limited to: instrument re-calibration, using freshly prepared calibration standards; replacement of reagent lots that give unacceptable blank values; instrument repair; substitution of backup instrumentation; sample data recalculation; or additional training. The need for these corrective actions is typically identified within a few days of the nonconformance event by the analyst or by their supervisor, and the corrective action is instituted immediately.

Long-Term Corrective Actions

Longer-term corrective action might include: instrumentation replacement; modification of data reduction algorithms; introduction of additional sample cleanup steps; personnel reassignment, if necessary, to achieve a better fit between analyst skills and method requirements. Such actions may be identified through operations review or through data quality audits. It may take several days to implement these types of corrective action, but it could also take several weeks. In the latter case, the ESE Laboratory Manager will contact the ESE QA Manager to determine whether analysis should continue or be put on hold, pending accomplishment of the corrective action.

With regard to report quality, corrective action is initiated at the time of the draft report review and might include: reformatting of tables or figures to ensure conformance to the QAPPs requirements and/or to make the data more understandable to the reader, reworking by senior

professional to be sure that the findings and conclusions presented verbally are supported by the data; or assignment of an editor to improve grammar, syntax, and punctuation.

Where corrective actions are needed, the following closed loop corrective action system is used:

- The problem is defined;
- Responsibility for investigating the problem is assigned;
- The cause of the problem is determined;
- The appropriate corrective action is determined;
- Responsibility for implementing the corrective action is assigned and accepted;
- Measures to assess the effectiveness of the corrective action are established;
- The corrective action is implemented; and,
- The effectiveness of the corrective action is verified.

Corrective actions for laboratory problems are specified in the laboratory SOPs. Documentation of corrective actions is recorded in logs maintained by the laboratory. Where problems effect sample processing or analysis, the corrective action is also included in the project supporting documentation.

13.3.1 Responsibilities

The ESE Laboratory Manager is responsible for reviewing the results of major corrective actions to determine and document the effectiveness of the actions in corrective action and follow-up memoranda. These memoranda are maintained in the filing system or QA records.

Laboratory staff have the responsibility to identify the need for corrective action on an on-going basis, communicating the need for corrective action, and documenting actions as required.

13.3.2 Project Specific Corrective Actions

Any laboratory corrective actions necessary to correct nonconformances with the QAPPs will be communicated by the ESE Laboratory Project Manager both verbally and in writing to the ESE Project Manager. The ESE Project Manager will notify the MD Project Manager in writing of nonconformance issues, who in turn will notify the MDNR RFI Project Coordinator.

13.4 Corrective Action During Data Validation and Data Assessment

The ESE Data Validator, the ESE QA Manager, or the various technical laboratory staff may identify the need for corrective action during either data validation or data assessment. Potential

types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (e.g. the holding time for samples is not exceeded, etc.). When the ESE Data Validator identifies a corrective action situation, the MD Project Manager will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions of this type will be documented by the ESE QA Manager.

14.0 Quality Assurance Reports to Management

The deliverables associated with the tasks identified in this QAPP and the accompanying RFI Workplan will contain separate QA sections in which data quality information collected during the task is summarized. The MD Project Manager will be responsible for these reports which will include data on the accuracy, precision, and completeness of the data, as well as the results of the performance and system audits, and any corrective action needed or taken during the project task.

14.1 Contents of Project Quality Assurance Reports

The QA reports will contain on a routine basis summaries of field and laboratory audits, summary information generated during the investigation reflecting on the achievement of specific data quality objectives, and a summary of corrective action that was implemented, and its immediate results on the project. Whenever necessary, updates on training provided and changes in key personnel, will be reported. All QA reports will be prepared by the MD Project Manager, or their designee including the ESE Project Manager or ESE QA Manager.

In the event of an emergency, or in case it is essential to implement corrective action immediately, QA reports can be made by telephone to the appropriate individuals, as identified in the Project Organization or Corrective Action sections of this QAPP; the MDNR RFI Project Coordinator will be one of the individuals notified. However, these events, and their resolution will be addressed thoroughly in the subsequent monthly status report for the Facility.

14.2 Frequency of Quality Assurance Reports

The QA Report will be prepared upon completion of the field and laboratory evaluation tasks. The frequency of any emergency reports that must be delivered verbally will be provided on an as-needed basis.

**Table 1-1. Target Analytical Constituents and Associated Detection Limits
McDonnell Douglas RFI, Hazelwood, Missouri Facility**

Constituent	Detection Limit (ug/kg, except as noted)
VOCs	
Acetone	10
1,2-Dichloroethylene	5
Perchloroethylene	5
Total Xylenes	5
PAHs	
Acenaphthene	330
Acenaphthylene	330
Anthracene	3.3
Benzo(a)anthracene	3.3
Benzo(a)pyrene	3.3
Benzo(b)fluoranthene	3.3
Benzo(g,h,i)perylene	3.3
Benzo(k)fluoranthene	3.3
Chrysene	3.3
Dibenzo(a,h)anthracene	3.3
Fluoranthene	3.3
Fluorene	70
Indeno(1,2,3-cd)pyrene	3.3
Naphthalene	330
Phenanthrene	3.3
Pyrene	3.3
Inorganics (mg/kg)	
Arsenic	5
Barium	1
Cadmium	0.5
Chromium	1
Lead	0.5
Mercury	0.02
Nickel	2
Selenium	0.5

Appendix A

ESE Laboratory QAPP

Laboratory Comprehensive Quality Assurance Plan
ENVIRONMENTAL SCIENCE & ENGINEERING, INC.
Peoria Laboratory

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September 1996

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LIST OF ACRONYMS AND ABBREVIATIONS

AAS	atomic absorption spectrophotometry
AIHA	American Industrial Hygiene Association
B	Cyanide, Total and Amenable to Chlorination (Free Cyanide)
BFB	bromofluorobenzene
BNAs	base/neutrals and acids
BOD	biochemical oxygen demand
BTEX	benzene, toluene, ethylbenzene, xylene
°C	degrees Celsius
CQAP	Comprehensive Quality Assurance Project Plan
CCC	calibration check compounds
CCS	continuing calibration standard
CCV	continuing calibration verification
CFR	Code of Federal Regulations
CLASS™	Chemical Laboratory Analysis and Scheduling System
CLP	Contract Laboratory Program
Cl-CH ₂ COOH	chloroacetic acid
COD	chemical oxygen demand
CVAA	mercury cold vapor atomic absorption
D	detection limit
DBCP	1,2-Dibromo-3-chloropropane
DFTPP	decafluorotriphenylphosphine
DBASE	data base
DHRS	Department of Health and Rehabilitative Services
DMR-QA	Discharge Monitoring Report - Quality Assurance
DI	deionized

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LIST OF ACRONYMS AND ABBREVIATIONS
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DO	dissolved oxygen
DOT	Department of Transportation
EC	Pesticides/PCBs
ECD	electron capture detector
EDB	1,2-dibromoethane
ELAP	Environmental Laboratory Approval Program
ELPAT	Environmental Lead Proficiency Analytical Testing
EPA	U.S. Environmental Protection Agency
ESE	Environmental Science & Engineering, Inc.
eV	electron volt
FLAA	flame atomic absorption
FID	flame ionization detector
FR	fraction code
FRN	file reference number
ft	foot
g	gram
g/kg	grams per kilogram
GC	gas chromatography
GC/FID	GC employing flame ionization detection
GC/HPLC	gas chromatography/high performance liquid chromatography
GC/MS	gas chromatograph/mass spectrometer
GC/MS/DS	gas chromatography/mass spectrometry/data system
GC/NPD	GC employing nitrogen-phosphorus detection
GFAA	graphite furnace atomic absorption
GLP	Good Laboratory Practice

LIST OF ACRONYMS AND ABBREVIATIONS
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GPC	gel permeation chromatography
H	sulfide
HCL	hydrochloric acid
HNO ₃	nitric acid
HPLC	high performance liquid chromatography
HWC	hazardous waste coordinator
H ₂ SO ₄	sulfuric acid
IC	ion chromatography
ICAP	inductively coupled argon plasma
ICB	initial calibration blank
ICS	interference check solution
ICV	initial calibration verification
ID	identification
IR	infrared
KCl	potassium chloride
kg	kilogram
KOH	potassium hydroxide
L	liter
LC	Laboratory Coordinator
LIMS	Laboratory Information Management System
MB	method blank
MBAS	methylene blue active substances
MDL	method detection limit
MS	Acid and Base/Neutral Extractables, PNAs, Nitroaromatics
MSDS	Material Safety Data Sheet

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LIST OF ACRONYMS AND ABBREVIATIONS
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MTBE	methyl-tert-butyl ether
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
mm ²	square millimeter
NaOH	sodium hydroxide
Na ₂ S ₂ O ₃	sodium thiosulfate
ng	nanogram
N	Metals, Hardness
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NTU	nephelometric turbidity unit
O	Oil and Grease, TRPH
PAT	Proficiency Analytical Testing Program
PAH	polynuclear aromatic hydrocarbons
PCB	polychlorinated biphenyl
PCP	pentachlorophenol
PQL	practical quantitation limit
% RSD	percent relative standard deviation
PID	photoionization device
PNA	polynuclear aromatic hydrocarbon
ppb	parts per billion
ppt	parts per thousand
psi	pounds per square inch

LIST OF ACRONYMS AND ABBREVIATIONS
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PVC	polyvinyl chloride
QA	quality assurance
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
QC	quality control
RF	response factor
RL	reportable detection limit
RP	replicate
RPD	relative percent difference
RSD	relative standard deviation
S	COD, TOC, Kjeldahl Nitrogen, Ammonia, Total Phosphorus
SD	serial dilution
SOP	standard operating procedure
SOW	Statement of Work
SP	standard spike/ laboratory control sample
SPCC	system performance check compound
SPM	sample matrix spike
SPX	analytical spike
SRT	sample receiving technician
SS	all solids (except VOCs)
STORET	storage and retrieval
SV	volatile solids
SUR	surrogate
THMS	trihalomethanes
TIC	tentatively identified compound
TOC	total organic carbon

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LIST OF ACRONYMS AND ABBREVIATIONS
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TOX	total organic halides
TRPH	total recoverable petroleum hydrocarbons
TSS	total suspended solids
$\mu\text{g/g}$	micrograms per gram
$\mu\text{g/L}$	micrograms per liter
μL	microliter
$\mu\text{mho/cm}$	micromhos per centimeter
UPS	United Parcel Service
USACE	U.S. Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USGS	U.S. Geological Survey
UV	ultraviolet
V	purgeable compounds
VOA	volatile organic aromatic compound
VOC	volatile organic compound
VP	purgeable aromatics (BTEX)
X	TOX
YSI	Yellow Springs Instruments
Z	total phenols

3.0 STATEMENT OF POLICY

3.1 QUALITY ASSURANCE (QA) STATEMENT OF POLICY

It is the policy of Environmental Science & Engineering, Inc. (ESE), Peoria Laboratory, to maintain an active quality assurance/quality control (QA/QC) program that provides analytical data of known and supportable quality and to ensure a high professional standard in analytical data generated in support of projects undertaken by the staff. An established QA/QC philosophy and program are essential for any organization to consistently produce valid laboratory data. To be valid, data is generated under controlled conditions which do not adversely affect data quality. Data is also interpreted by capable professionals who are trained in appropriate scientific disciplines, maintain a current knowledge of their field, and are experts in the applications for which the data is used. The objectives of the QA/QC program are to estimate the quality of each analytical system including precision, accuracy, and sensitivity sufficient for each project. The QA/QC program also assists in the early recognition of nonconformances which might affect data quality. ESE supports a corporate-wide Quality Education System (QES). All employees are trained in the quality improvement process. The training is supplemented at the department level by instructing employees on the importance of QA/QC and the price of nonconformance.

3.2 SCOPE

This Comprehensive Quality Assurance Plan (CQAP) applies to the analyses of samples received by the Peoria Laboratory. The Peoria Laboratory provides field sample collection when required. In addition, the Peoria Laboratory works with field sampling personnel to ensure that all samples received were collected, preserved, and delivered to the laboratory such that the quality of the analytical results are not adversely affected. All major environmental studies and analyses conducted by ESE Peoria Laboratory for projects under the guidance of client or state/federal government agencies are performed in accordance with this CQAP.

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When appropriate, this CQAP is filed with a client and/or regulatory agency, and once approved, is referenced in lieu of the repetitive submission of plans for which only a portion of the information is changed.

3.3 DOCUMENT CONTROL

This CQAP is revised periodically as procedural changes become necessary. Changes are documented by the date of each section. The Peoria Laboratory QA/QC Department keeps a distribution list and assigns a unique number to each copy of the CQAP. When a section is revised, the revision date replaces the original date in the heading code and the table of contents is updated. Copies of the revised sections are provided to each individual on the distribution list.

These procedures apply once the plan has been finalized and implemented. These procedures do not apply to draft documents.

4.0 ORGANIZATION AND RESPONSIBILITIES

4.1 LABORATORY OPERATIONS CAPABILITIES

ESE laboratory operations include the following capabilities:

1. Groundwater and surface water analysis,
2. Soil and sediment analysis,
3. Wastewater analysis,
4. Drum analysis,
5. Tissue analysis, and
6. Underground storage tank analysis.

4.2 LABORATORY OPERATIONS PERSONNEL

The organizational structure and areas of responsibility for the Peoria laboratory are shown on the organizational chart in Figure 4-1. Brief descriptions of the major duties and responsibilities of the key laboratory positions as shown on the organizational chart are:

4.2.1 Laboratory Director

The Laboratory Director provides budgetary oversight of laboratory operations to verify that required financial controls and accounting procedures are in place. The Laboratory Director formulates long-term goals in marketing, facilities, staffing, equipment, and analytical capabilities. The Laboratory Director is responsible for the overall management of the analytical laboratory, including the appointment and supervision of the Laboratory Information Services Manager, Laboratory Operations Manager, Customer Services Manager, and Laboratory Quality Assurance Manager.

4.2.2 Laboratory Quality Assurance Manager

The Laboratory QA Manager is responsible for the oversight of the quality assurance program and auditing its operational execution, directing quality issue resolution and assuring the implementation of suitable corrective action. In addition, the QA Manager coordinates certifications and other recognitions of the laboratory's proficiency by outside agencies and companies, and provides technical guidance on all quality activities.

4.2.3 Laboratory Information Services Manager

The Laboratory Information Services Manager oversees the Peoria Laboratory's computerized data management system and is responsible for maintaining ESE's Chemical Laboratory Analysis Scheduling System (CLASS™), for approval of all changes made to CLASS™, ensuring that regular backups are performed, observing all security procedures, implementing new software, and general maintenance.

4.2.4 Laboratory Operations Manager

The Laboratory Operations Manager is responsible for the scheduling and management of daily laboratory operations and the ongoing effective implementation of appropriate quality control measures. The operations manager provides technical guidance, assures staff is suitably qualified and trained, and makes recommendations concerning staffing, facilities, instrumentation/equipment, and quality program enhancements.

4.2.5 Customer Services Manager

The Customer services manager is responsible for the overall management of the project operations within the laboratory including the appointment and supervision of the Laboratory Project Managers.

4.2.6 Laboratory Project Managers

The Laboratory Project Managers are responsible for the overall management of project operations within the Peoria Laboratory. The Project Managers act as liaisons between clients and laboratory operations and are responsible for coordination of sample analyses to meet project or client objectives, overseeing report preparation and reviewing project data for completeness, accuracy and compliance to project requirements. The project managers communicate project changes to the appropriate laboratory staff and keep the client informed concerning the status of their project(s).

4.2.7 Sample Custodian

The Sample Custodian checks in the samples from clients upon receipt by the laboratory. The Sample Custodian compares all samples contained in the shipment to the Chain-of- Custody sheets to ensure that all samples designated on the logsheet have been received. The Sample Custodian will note any special remarks concerning the shipment, log all samples into the Laboratory Information Management System (CLASS™), and deliver the logsheets (Arrival Notices) to the Project Managers, and Laboratory Department Managers. The Sample Custodian places samples in appropriate storage areas.

4.2.8 Laboratory Department Managers

The Laboratory Department Managers of Inorganics, Extractions, Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC), and Gas Chromatography/Mass Spectrometry (GC/MS) are responsible for the daily operations of their respective sections. The managers' duties include assuring employees are properly trained, instruments/equipment are properly calibrated and maintained, all necessary SOPs are available and up-to-date, and documentation is suitably recorded and complete. In addition, laboratory managers confirm that

projects and quality control are performed as per clients's requirements and that corrective action is promptly taken to resolve identified quality issues.

4.2.9 Laboratory Analysts

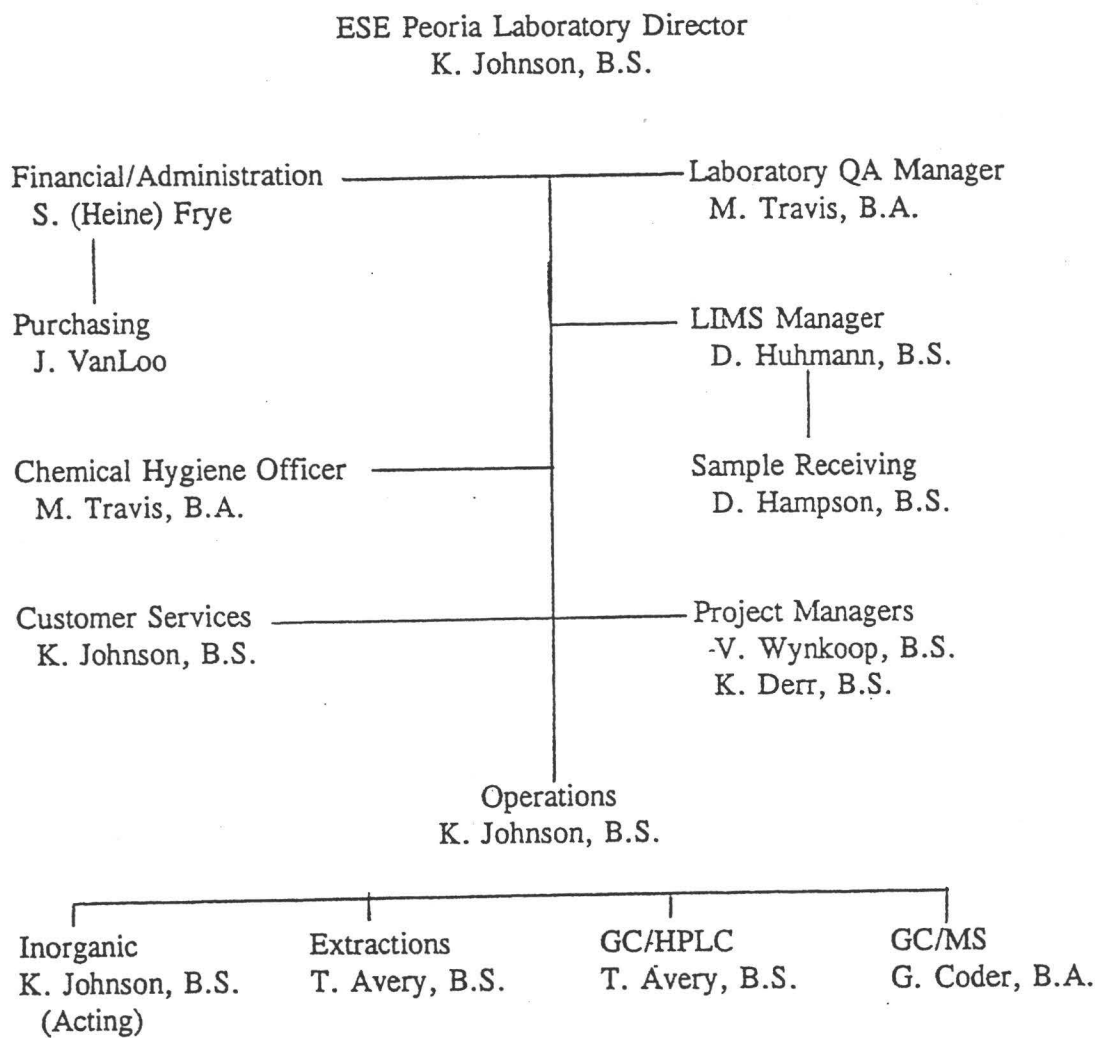
Laboratory Analysts are responsible for the application of the correct SOPs using laboratory techniques and instrumentation and quality control to produce valid data which meet or exceed the client's requirements.

4.2.1.10 Laboratory Chemical Hygiene Officer

The Chemical Hygiene Officer (CHO) assists laboratory supervisors in implementing the Chemical Hygiene Program. The CHO will provide for Chemical Hygiene Training for analysts, review laboratory safety manual and SOPs, perform safety audits of the laboratory and perform inspections of laboratory safety equipment to determine compliance. Areas of non-compliance will be reported to the appropriate manager. The CHO will evaluate worker chemical exposure and will provide a written report of each exposure assessment or determination to the Laboratory Director for action as necessary.

The CHO maintains an inventory of all radioactive sources within the Peoria Laboratory.


Figure 4-1 ESE PEORIA LABORATORY ORGANIZATION CHART



Comprehensive Quality Assurance Plan

for
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9-6-96

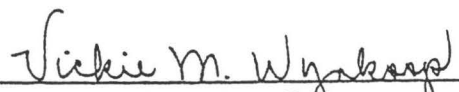
Date



Michael A. Travis, B.A.
Laboratory QA Manager
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9-6-96

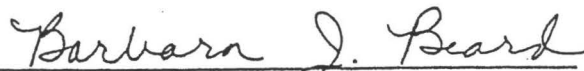
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Date

5.0 QA OBJECTIVES FOR MEASUREMENT DATA

5.1 LABORATORY ANALYSIS

Analyses are performed according to standard U.S. Environmental Protection Agency (EPA) analytical procedures for analysis of water and soil/sediment unless otherwise specified (Tables 5-1 through 5-55). EPA precision and accuracy data and ESE Laboratory analytical experience were used as the basis for developing criteria to assess laboratory method performance as noted. These limits are subject to change based on actual historic and current performance; updates are provided for insertion into all copies of QAPPs, as appropriate. Limits are updated on a yearly basis unless otherwise specified. Specific compounds are used for controlling purposes in multianalyte methods and are identified in Tables 5-2 through 5-54. Laboratory method performance is evaluated and controlled using calibration checks, blanks, and QC check samples; sample accuracy and precision are evaluated using sample duplicate data, matrix spike, and matrix spike duplicate data. ESE's method control procedures are discussed in Section 11.

The reportable detection limits (RLs) achievable for all parameters are listed in Tables 5-3 through 5-55 (odd numbered tables). The RLs are values, above the method detection limit, which are reported with confidence for typical environmental matrices. The reportable detection limits are not method detection limits (MDLs). Method detection limits are discussed in Section 11. The RLs for waters and those calculated for solids are typically reported as listed, if no matrix and/or other interferences (e.g. salt water) are found to be present (subject to adjustment for dilutions and/or moisture contents).

The following is a brief explanation of the terms and organic method footnotes that appear in Tables 5-1 through 5-57. When recovery criteria was not listed in the method and historical data was not available, the laboratory set achievable QC criteria goals; as noted.

Reference: The reference of the standard analytical methodology used for each procedure.

Precision: Evaluated based on the relative percent difference (RPD) of duplicate spikes (see Section 11 for definition).

Accuracy: Evaluated based on the percent recovery of each spike (see Section 11 for definition).

Units: Volume in liters (L) [e.g., micrograms per liter ($\mu\text{g/L}$)] indicates a water matrix; control spikes are added to organic-free laboratory water. Mass in grams (g) or kilograms (kg) [e.g., milligrams per kilogram (mg/kg)] indicates a soil/sediment matrix; control spikes are added to blank sample matrices, blank soil, or organic-free laboratory water, depending on the analytical procedure.

Organic Method Footnotes:

- a Matrix spike and QC check sample compound.
- b Accuracy and precision based on method criteria, unless otherwise noted.
- c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
- d Appendix IX compounds.
- e Compound analysis available upon request.
- f Compound not listed in method.
- g Surrogate compound.
- h Estimated detection limits listed in method times a factor of ten.
- i Criteria adopted from USEPA Contract Laboratory Program Statement of Work, March 1990.

Table 5-1. Sample Preparation Methods for U.S. EPA SW846 Methods

Sample Preparation Method Number	Description	Matrix	Sample Preparation for Methods
EPA 3005	Acid Digestion	Aqueous	EPA 6010
EPA 3010	Acid Digestion	Aqueous	EPA 6010
EPA 3020	Acid Digestion	Aqueous	EPA 7041, 7060, 7131, 7421, 7740, 7841
EPA 3050	Acid Digestion	Solid	EPA 6010, 7041, 7060, 7131, 7421, 7740, 7841
EPA 3510	Separator Funnel Liquid- Liquid Extraction	Aqueous	EPA 8080, 8141, 8270, 8310
EPA 3520	Continuous Liquid- Liquid Extraction	Aqueous	EPA 8080, 8141, 8270, 8310
EPA 3540	Soxhlet Extraction	Solid	EPA 8080, 8141, 8270, 8310
EPA 3550	Sonication Extraction	Solid	EPA 8080, 8141, 8270, 8310
EPA 5030	Purge-And-Trap	Aqueous, Solid	EPA 8010, 8020, 8240, 8260
EPA 3630	Silica Gel Cleanup	Aqueous, Solid	EPA 8080
EPA 3640	Gel-Permeation Cleanup	Aqueous, Solid	EPA 8080, 8141, 8270
EPA 3660	Sulfur Cleanup	Aqueous, Solid	EPA 8080

Source: ESE.

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Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, Oil and Grease, TRPH, and TOX Analyses

Parameter	Units	Reference	Method Criterion *	
			Precision (Max RPD)	Accuracy (Percent Recovery)
Aluminum, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Aluminum, Solid	mg/kg	EPA 3050, 6010	20	80-120
Antimony, Total ^b	µg/L	EPA 204.2, 3020, 7041	20	80-120
Antimony, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Antimony, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Antimony, Solid ^b	mg/kg	EPA 3050, 7041	20	80-120
Arsenic, Total ^b	µg/L	EPA 206.2, 200.7, 3005, 3010, 6010, 3020, 7060	20	80-120
Arsenic, Solid ^b	mg/kg	EPA 3050, 7060, 6010	20	80-120
Barium, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Barium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Beryllium, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Beryllium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Cadmium, Total ^b	µg/L	EPA 213.2, 3020, 7131	20	80-120
Cadmium, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Cadmium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Cadmium, Solid ^b	mg/kg	EPA 3050, 7131	20	80-120
Calcium, Total ^b	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Calcium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Chromium, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Chromium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Cobalt, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Cobalt, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses
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Parameter	Units	Reference	Method Criterion *	
			Precision (Max RPD)	Accuracy (Percent Recovery)
Copper, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Copper, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Iron, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Iron, Solid	mg/kg	EPA 3050, 6010	20	80-120
Lead, Total ^b	µg/L	EPA 239.2, 3020, 7421	20	80-120
Lead, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Lead, Solid ^b	mg/kg	EPA 3050, 6010,	20	80-120
Lead, Solid ^b	mg/kg	EPA 3050, 7421	20	80-120
Magnesium, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Magnesium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Manganese, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Manganese, Solid	mg/kg	EPA 3050, 6010	20	80-120
Mercury, Total ^b	µg/L	EPA 245.1, 7470	20	80-120
Mercury, Solid ^b	mg/kg	EPA 7471	20	80-120
Molybdenum, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Molybdenum, Solid	mg/kg	EPA 3050, 6010	20	80-120
Nickel, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Nickel, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Potassium, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Potassium, Solid	mg/kg	EPA 3050, 6010	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses
(Continued, Page 3 of 7)

Parameter	Units	Reference	Method Criterion ^a	
			Precision (Max RPD)	Accuracy (Percent Recovery)
Selenium, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Selenium, Total ^b	µg/L	EPA 270.2, 3020, 7740	20	80-120
Selenium, Solid ^b	mg/kg	EPA 3050, 7740	20	80-120
Selenium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Silicon, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Silicon, Solid	mg/kg	EPA 3050, 6010	20	80-120
Silver, Total ^d	µg/L	EPA 272.2	20	54-125
Silver, Total ^{b,d}	µg/L	EPA 200.7, 3005, 3010, 6010	20	54-125
Silver, Solid ^{b,d}	mg/kg	EPA 3050, 6010	20	54-125
Sodium, Total	mg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Sodium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Strontium, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Strontium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Thallium, Total ^b	µg/L	EPA 279.2, 3020, 7841	20	80-120
Thallium, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Thallium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Thallium, Solid ^b	mg/kg	EPA 3050, 7841	20	80-120
Titanium, Total	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Titanium, Solid	mg/kg	EPA 3050, 6010	20	80-120
Tin, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Tin, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Vanadium, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Vanadium, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120
Zinc, Total ^b	µg/L	EPA 200.7, 3005, 3010, 6010	20	80-120
Zinc, Solid ^b	mg/kg	EPA 3050, 6010	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 4 of 7)

Parameter	Units	Reference	Method Criterion *	
			Precision (Max RPD)	Accuracy (Percent Recovery)
Acidity, Total	mg/L-CaCO ₃	EPA 305.1	20	80-120
Alkalinity, Total	mg/L-CaCO ₃	EPA 310.1	20	80-120
BOD, 5-day	mg/L	EPA 405.1	20	80-120
cBOD	mg/L	EPA 405.1	20	80-120
Bromide	mg/L	EPA 320.1, EPA 300, 9056	20	80-120
BTU	Cal/lb	ASTM D-240	N/A	N/A
Carbon, Total	mg/L	EPA 415.2, 9060	20	80-120
Carbon, TOC	mg/L	EPA 415.2, 9060	20	80-120
Carbon, TOC, Solid	mg/kg	EPA 9060 (Mod)	20	80-120
Carbon, Percent Content	% Organic	Walkley Black	N/A	N/A
COD	mg/L	EPA 410.4	20	80-120
Chloride	mg/L	EPA 325.3, 9252, SM 407C	20	80-120
Chloride	mg/L	EPA 300, 9056	20	80-120
Chlorine, Percent	% Chlorine	ASTM D-808	N/A	N/A
Chlorine, Total Residual	mg/L	EPA 330.5	20	80-120
Chromium (+6)	mg/L	EPA 7196	20	80-120
Chromium (+6), Solid	mg/kg	EPA 3060, 7196	20	80-120
Color	Color Units	EPA 110.2	N/A	N/A
Corrosivity	mm/yr	EPA 9040, 1110	N/A	N/A
Cyanide ^b	mg/L	EPA 335.2, 9010	20	80-120
Cyanide, Solid ^b	mg/kg	EPA 9010 (Mod)	20	80-120
Cyanide, Ammenable ^b	mg/L	EPA 335.1	20	80-120
Dissolved Oxygen	mg/L	EPA 360.1	20	N/A
Fluoride	mg/L	EPA 340.2	20	80-120
Fluoride	mg/L	EPA 300, 9056	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 5 of 7)

Parameter	Units	Reference	Method Criterion *	
			Precision (Max RPD)	Accuracy (Percent Recovery)
Hardness	mg/L-CaCO ₃	EPA 200.7, 130.2	20	80-120
Ignitability	°C	EPA 1010	N/A	N/A
MBAS (foaming agents)	mg/L	EPA 425.1	20	80-120
Nitrogen, NO ₂ + NO ₃	mg/L-as N	EPA 353.2	20	80-120
Nitrogen, NO ₂ + NO ₃	mg/kg-as N	EPA 353.2 (Mod)	20	80-120
Nitrogen, NO ₃ ^c	mg/L-as N	EPA 300, 9056	20	80-120
Nitrogen, NO ₃ ^c	mg/L-as N	EPA 353.2	20	80-120
Nitrogen, NO ₂	mg/L-as N	EPA 300, 9056	20	80-120
Nitrogen, NO ₂	mg/L-as-N	EPA 353.2	20	80-120
Nitrogen, NH ₃ + NH ₄	mg/L-as N	EPA 350.3	20	80-120
Nitrogen, TKN	mg/L-as N	EPA 351.4	20	80-120
Nitrogen, TKN, Solid	mg/kg-as N	EPA 351.4	20	80-120
Odor, 25°C	Thrsh No	EPA 140.1	N/A	N/A
Oil and Grease, Grav	mg/L	EPA 413.1	20	80-120
Oil and Grease, IR	mg/L	EPA 413.2	20	80-120
Oil and Grease, IR, Solid	mg/kg	EPA 9071	20	80-120
Percent Moisture	% Wet Weight	EPA 160.3	20	N/A
Percent Solids	% Dry Weight	EPA 160.3	20	N/A
pH (including solids)	Std Units	EPA 150.1, 9040	20	N/A
Phenols	mg/L	EPA 420.1	20	80-120
Phenols, Solid	mg/kg	EPA 9065	20	80-120
Phosphorus, Total	mg/L-as P	EPA 365.2	20	80-120
Phosphorus, Ortho	mg/L-as P	EPA 300, 9056	20	80-120
Phosphorus, Ortho	mg/L-as P	EPA 365.2	20	80-120

Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 6 of 7)

Parameter	Units	Reference	Method Criterion ^a	
			Precision (Max RPD)	Accuracy (Percent Recovery)
Residue, Settleable	mg/L	EPA 160.5	20	N/A
Residue, Susp. (TSS)	mg/L	EPA 160.2	20	N/A
Residue, Diss., Total (TDS) 105°C	mg/L	EPA 160.1	20	N/A
Residue, Total (TS)	mg/L	EPA 160.3	20	N/A
Residue, Volatile	mg/L	EPA 160.4	20	N/A
Petroleum hydrocarbons (TRPH)	mg/L	EPA 418.1	20	80-120
Petroleum hydrocarbons, Solid	mg/kg	EPA 9071/418.1	20	80-120
Silica	mg/L	EPA 200.7	20	80-120
Specific Cond.	μmhos/cm	EPA 120.1	20	N/A
Sulfate	mg/L	EPA 300, 9056	20	80-120
Sulfate	mg/L	EPA 375.4, 9038	20	80-120
Sulfide ^b	mg/L	EPA 376.2, 9030	20	80-120
Sulfide, Solid ^b	mg/kg	EPA 9030	20	80-120
Sulfite	mg/L	EPA 377.1	20	80-120
Sulfur, Percent	% Sulfur	ASTM D-129	N/A	N/A
Temperature	°C	SM 2550 B	N/A	N/A
TOX	μg/L-Cl	EPA 9020A	20	80-120
TOX, Solid	mg/kg	EPA 9020A	20	80-120
Turbidity	NTU	EPA 180.1	20	N/A
TCLP	--	EPA 1311	N/A	N/A

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Table 5-2. Summary of Precision and Accuracy Criteria for Inorganics Analysis, Metals Analysis, and Oil and Grease, TRPH, and TOX Analyses (Continued, Page 7 of 7)

Note:

CLP = EPA Contract Laboratory Program.
N/A = not applicable.
SOW = statement of work.
TCLP = toxicity characteristics leaching procedure.
TOX = total organic halides.
TRPH = total recoverable petroleum hydrocarbons.

References:

ASTM D2974--American Society for Testing and Materials Designation: D2974-87, July 1987.
EPA 100-400--Methods for Chemical Analyses of Water and Waste. EPA 600/4-79-20--Revised March 1983.
EPA 1310-9073--Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition (Method 9073, draft 1989: oil and grease methods exclude 7.8 and 7.10).
SM 4500-N--Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

- ^a All precision and accuracy criteria is referenced from EPA CLP SOW 3/90.
- ^b Appendix IX compounds.
- ^c NO_3 (as N) by EPA 353.2 is calculation of $(\text{NO}_2 + \text{NO}_3) - (\text{NO}_2)$; also, method criteria do not apply.
- ^d The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses

Parameter	Reference	Reporting Limit	
		Aqueous ^a (µg/L)	Solid ^b (mg/kg)
Aluminium	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Antimony	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Antimony	EPA 204.2, 3020, 3050, 7041	10	1.0
Arsenic	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Arsenic	EPA 206.2, 3020, 3050, 7060	10	1.0
Barium	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Beryllium	EPA 200.7, 3005, 3010, 3050, 6010	5.0	0.5
Cadmium	EPA 200.7, 3005, 3010, 3050, 6010	5.0	0.5
Cadmium	EPA 213.2, 3020, 3050, 7131	0.2	0.02
Calcium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Chromium	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Cobalt	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Copper	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Iron	EPA 200.7, 3005, 3010, 3050, 6010	100	10
Lead	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Lead	EPA 239.2, 3020, 3050, 7421	5.0	0.5
Magnesium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Manganese	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Mercury	EPA 245.1, 7470, 7471	0.2	0.02
Molybdenum	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0

Table 5-3.

Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH,
and TOX Analyses (Continued, Page 2 of 5)

Parameter	Reference	Reporting Limit	
		Aqueous ^a ($\mu\text{g/L}$)	Solid ^b (mg/kg)
Nickel	EPA 200.7, 3005, 3010, 3050, 6010	20	2.0
Potassium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Selenium	EPA 200.7, 3005, 3010, 3050, 6010	75	7.5
Selenium	EPA 270.2, 7740, 3020, 3050	5.0	0.5
Silicon	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Silver	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Silver	EPA 272.2	0.5	0.05
Sodium	EPA 200.7, 3005, 3010, 3050, 6010	500	50
Strontium	EPA 200.7, 6010, 3005, 3010, 3050	10	1.0
Thallium	EPA 200.7, 3005, 3010, 3050, 6010	100	10
Thallium	EPA 279.2, 3020, 3050, 7841	10	1.0
Tin	EPA 200.7, 3005, 3010, 3050, 6010	100	10
Titanium	EPA 200.7, 3005, 3010, 3050, 6010	50	5.0
Vanadium	EPA 200.7, 3005, 3010, 3050, 6010	10	1.0
Zinc	EPA 200.7, 3005, 3010, 3050, 6010	20	2.0

Parameter (Inorganic) Units		Reference	Reporting Limit ^c
Acidity, Total	mg/L-CaCO ₃	EPA 305.1	2.0
Alkalinity, Total	mg/L-CaCO ₃	EPA 310.1	5.0
BOD, 5-day	mg/L	EPA 405.1	1.0
cBOD	mg/L	EPA 405.1	1.0
Bromide	mg/L	EPA 320.1, EPA 300, 9056	0.10
BTU	Cal/lb	ASTM D-240	100
Carbon, Total	mg/L	EPA 415.2, 9060	1.0
Carbon, TOC	mg/L	EPA 415.2, 9060	1.0

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses
(Continued, Page 3 of 5)

Parameter	Units	Reference	Reporting Limit ^a
Carbon, TOC, Solid	mg/kg	EPA 9060 (Mod)	100
Carbon, Percent	% Organic	Walkley Black	0.1
COD	mg/L	EPA 410.4 (Mod)	5.0
Chloride	mg/L	EPA 325.3, 9252, SM 407C	5.0
Chloride	mg/L	EPA 300, 9056	0.5
Chlorine, Percent	% Chlorine	ASTM-D 808	0.1
Chlorine, Total Residual	mg/L	EPA 330.5	0.05
Chromium (+6)	mg/L	EPA 7196	0.05
Chromium (+6) Solid	mg/kg	EPA 3060, 7196	5.0
Color	Color Units	EPA 110.2	N/A
Corrosivity	mm/yr	EPA 9040, 1110	N/A
Cyanide	mg/L	EPA 335.2, 9010	0.005
Cyanide, Solid	mg/kg	EPA 9010 (Mod)	0.50
Cyanide, Ammenable	mg/L	EPA 335.1	0.005
Dissolved Oxygen	mg/L	EPA 360.1	N/A
Fluoride	mg/L	EPA 340.2	0.1
Fluoride	mg/L	EPA 300, 9056	0.5
Hardness	mg/L-CaCO ₃	EPA 200.7, 130.2	5.0
Ignitability	°C	EPA 1010	N/A
MBAS (foaming agents)	mg/L	EPA 425.1	0.2
Nitrogen, NO ₂ + NO ₃	mg/L-as N	EPA 353.2	0.10
Nitrogen, NO ₂ + NO ₃	mg/kg-as N	EPA 353.2 (Mod)	10
Nitrogen, NO ₃	mg/L-as N	EPA 300, 9056	0.01
Nitrogen, NO ₃	mg/L-as N	EPA 353.2	0.10

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Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses
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Parameter	Units	Reference	Reporting Limit ^a
Nitrogen, NO ₂	mg/L-as N	EPA 300, 9056	0.01
Nitrogen, NO ₂	mg/L-as N	EPA 353.2	0.05
Nitrogen, NH ₃ + NH ₄	mg/L-as N	EPA 350.3	0.1
Nitrogen, TKN	mg/L-as N	EPA 351.4	0.1
Nitrogen, TKN, Solid	mg/kg-as N	EPA 351.4	10
Odor, 25°C	Thrsh No	EPA 140.1	N/A
Oil and Grease, Grav	mg/L	EPA 413.1	5.0
Oil and Grease, IR	mg/L	EPA 413.2	1.0
Oil and Grease, IR Solid	mg/kg	EPA 9071	10
Percent Moisture	% Wet Weight	EPA 160.3	N/A
Percent Solids	% Dry Weight	EPA 160.3	N/A
pH (including solids)	Std Units	EPA 150.1, 9040	N/A
Phenols	mg/L	EPA 420.1	0.05
Phenols, Solid	mg/kg	EPA 9065	1.0
Phosphorus, Total	mg/L-as P	EPA 365.2	0.05
Phosphorus, Ortho	mg/L-as P	EPA 365.2	0.05
Phosphorus, Ortho	mg/L-as P	EPA 300, 9056	0.05
Residue, Settleable	mg/L	EPA 160.5	0.1
Residue, Susp. (TSS)	mg/L	EPA 160.2	1.0
Residue, Diss., Total (TDS) 105°C	mg/L	EPA 160.1	1.0
Residue, Total (TS)	mg/L	EPA 160.3	1.0
Residue, Total Volatile	mg/L	EPA 160.4	1.0
Petroleum Hydrocarbons (TRPH)	mg/L	EPA 418.1	1.0

Table 5-3. Reporting Limit Data for Metals, Inorganics, Oil and Grease, TRPH, and TOX Analyses
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Parameter	Units	Reference	Reporting Limit ^c
Petroleum Hydrocarbons, Solid	mg/kg	EPA 9071/418.1	10
Silica	mg/L	EPA 200.7	2.0
Specific Cond.	$\mu\text{mho/cm}$	EPA 120.1	10
Sulfate	mg/L	EPA 300, 9056	0.5
Sulfate		EPA 375.4, 9038	5.0
Sulfide	mg/L	EPA 376.2, 9030	0.2
Sulfide, Solid	mg/kg	EPA 9030	5.0
Sulfite	mg/L	EPA 377.1	2.0
Sulfur, Percent	% Sulfur	ASTM D-129	0.1
Temperature	°C	EPA 170.1	N/A
TOX	$\mu\text{g/L-Cl}$	EPA 9020A	5.0
TOX, Solid	mg/kg	EPA 9020A	10
Turbidity	NTU	EPA 180.1	0.1

Note: $\mu\text{g/L}$ = micrograms per liter.
 mg/kg = milligrams per kilogram.
 mg/L = milligrams per liter.
NTU = nephelometric turbidity unit.
 $\mu\text{mho/cm}$ = microhms per centimeter.

- ^a Based on ESE's instrument detection limit (IDL) studies unless indicated differently. The EPA Contract Laboratory Program (CLP) SOW 3/90 requirements are followed when the IDL studies are conducted.
- ^b Based on aqueous IDL studies times a factor of 0.1 to take into account sample weight and final volume of digestate, unless indicated differently.
- ^c Based on the lowest standard that ESE routinely uses. For solids, the reporting limits are adjusted for sample weight and final volume.

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Table 5-4. Analytes, Precision, and Accuracy Data For Volatile Organics, EPA 502.2

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
Chloroform ^a	33	77-143
Bromodichloromethane ^a	33	79-137
Dibromochloromethane ^a	33	23-125
Bromoform ^a	33	43-106
THMs, total ^{a,c}	33	62-128

Reference: EPA Method 502.2 - Volatile Halogenated Compounds in Water by Purge and Trap Gas Chromatography, USEPA, (Revision 2.0) 1989.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-5. Reporting Limit Data For Volatile Organics, EPA 502.2

Parameter	<u>Reporting Limits</u>
	Aqueous ($\mu\text{g/L}$)
Chloroform*	1.0
Bromodichloromethane*	1.0
Dibromochloromethane*	1.0
Bromoform*	1.0
THMs, total*	4.0

* Matrix spike and QC check sample compound.

Source: ESE.

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Table 5-6. Analytes, Precision, and Accuracy Data For EDB and DBCP, EPA 504

	<u>Aqueous^b</u>	
	Precision (RPD)	Accuracy (%Recovery)
1,2-Dibromoethane (EDB) ^{a,d}	20	60-140
DBCP (nemagon) ^{a,d}	30	60-140

Reference: EPA Method 504- 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-chloropropane in Water by Microextraction and Gas Chromatography, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision criteria based on method criteria, unless otherwise noted.

^d Appendix IX compounds.

Source: ESE.

Table 5-7. Reporting Limit Data for EDB and DBCP, EPA 504

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
1,2-Dibromoethane (EDB) ^{a,d}	0.05
DBCP (nemagon) ^{a,d}	0.10

^a Matrix spike and QC check sample compound.

^d Appendix IX compounds.

Source: ESE.

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Table 5-8. Analytes, Precision, and Accuracy Data For Organohalide Pesticides and Aroclors, EPA 505

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
Hexachlorobenzene ^a	19	64-144
Hexachlorocyclopentadiene ^a	22	38-108

Reference: EPA Method 505 - Analysis of Organohalide Pesticides and Commercial Polychlorinated Biphenyl (PCB) Products in Water by Microextraction and Gas Chromatography, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

Table 5-9. Reporting Limits Data for Organohalide Pesticides and Aroclors, EPA 505

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
Hexachlorobenzene*	0.20
Hexachlorocyclopentadiene*	0.20

* Matrix spike and QC check sample compound.

Source: ESE.

Table 5-10. Analytes, Precision, and Accuracy Data For Phthalate and Adipate Esters, EPA 506

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
Dimethylphthalate	66	25-121
Diethyl phthalate	68	23-119
Di-N-butyl phthalate	66	23-113
Butylbenzyl phthalate	63	26-116
bis (2-Ethylhexyl) adipate ^a	78	15-123
bis (2-Ethylhexyl) phthalate ^{a,c}	78	15-130
Di-N-octyl phthalate ^c	78	15-131

Reference: EPA Method 506 - Determination of Phthalate and Adipate Esters in Drinking Water by Liquid-Liquid Extraction and Gas Chromatography with Photoionization Detection, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-11. Reporting Limits Data For Phthalate and Adipate Esters, EPA 506

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
Dimethylphthalate	5.0
Diethyl phthalate	5.0
Di-N-butyl phthalate	5.0
Butylbenzyl phthalate	5.0
bis (2-Ethylhexyl) adipate ^a	5.0
bis (2-Ethylhexyl) phthalate ^a	5.0
Di-N-octyl phthalate	10

^a Matrix spike and QC check sample compound.

Source: ESE.

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Table 5-12. Analytes, Precision, and Accuracy Data For Nitrogen and Phosphorous Containing Pesticides, EPA 507

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (%Recovery)
Alachlor ^a	34	62-128
Atrazine ^{a,c}	26	70-130
Bromacil	30	64-118
Butachlor ^a	12	80-112
Butylate	65	34-160
Cyanazine ^{c,f}	30	70-130
Diazinon	18	92-138
Dyfonate ^{c,f}	20	80-120
EPTC	32	58-112
Malathion ^{c,f}	20	80-120
Metolachlor ^{a,c}	30	70-130
Metribuzin ^{a,c}	30	70-130
Phorate ^{c,f}	20	80-120
Propazine	26	68-116
Simazine ^a	21	79-121
Terbufos	12	85-109
Trifluralin ^{c,f}	30	50-150
2-NMX ^a	N/A	52-115

Table 5-12. Analytes, Precision, and Accuracy Data For Nitrogen and Phosphorous Containing Pesticides, EPA 507 (Continued, Page 2 of 2)

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)

Reference: EPA Method 507 - Determination of Nitrogen- and Phosphorous-containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorous Detector, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

- ^a Matrix spike and QC check sample compound.
- ^b Accuracy and precision based on method criteria, unless otherwise noted.
- ^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
- ^f Compound not listed in method.
- ^z Surrogate compound.

Source: ESE.

Table 5-13. Reporting Limit Data for Nitrogen and Phosphorus Containing Pesticides, EPA 507

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
Alachlor ^a	0.5
Atrazine ^a	0.5
Bromacil ^b	2.5
Butachlor ^{a,h}	3.8
Butylate	1.5
Cyanazine ^f	1.0
Diazinon	2.5
Dyfonate ^f	0.5
EPTC	2.5
Malathion ^f	0.5
Metolachlor ^{a,h}	7.5
Metribuzin ^{a,h}	1.5
Phorate ^f	0.5
Propazine ^b	1.3
Simazine ^{a,h}	0.75
Terbufos	5.0
Trifluralin ^f	0.5

^a Matrix spike and QC check sample compound.

^f Compound not listed in method.

^b Estimated detection limits listed in EPA Method 507, times ten.

Source: ESE.

Table 5-14. Analytes, Precision, and Accuracy Data For Chlorinated Pesticides, EPA 508

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
BHC,A	33	62-122
BHC,G (Lindane) ^a	33	60-118
Heptachlor ^a	36	63-133
Endosulfan I	30	61-113
Dieldrin ^a	30	61-113
Endrin ^a	30	62-114
DDD	18	88-126
DDT ^a	45	62-162
Methoxychlor ^a	39	64-146
BHC,B	39	64-146
BHC,D	33	58-114
Aldrin ^a	33	58-114
Heptachlor epoxide ^a	30	61-113
Hexachlorobenzene ^a	66	34-164
Chlordane, G	36	63-135
Chlordane, A	36	63-135
DDE	36	63-135
Endosulfan II	33	62-122
Endrin aldehyde	27	64-112
Endosulfan sulfate	45	56-148
Endrin ketone ^{c,e,f}	30	50-150
Propachlor ^a	27	75-131
Trifluralin ^{c,e}	30	70-130

Table 5-14. Analytes, Precision, and Accuracy Data For Chlorinated Pesticides, EPA 508
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	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
Toxaphene	20	80-120
Chlordane, Technical	20	80-120
TCX ^{c,e}	N/A	52-127
DCB ^{c,e}	N/A	47-148
Aroclor 1016	30	70-130
Aroclor 1254	30	70-130
Aroclor 1260	30	70-130

Reference: EPA Method 508 - Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

- ^a Matrix spike and QC check sample compound.
- ^b Accuracy and precision based on method criteria, unless otherwise noted.
- ^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
- ^d Compound analysis available upon request.
- ^e Compound not listed in the method.
- ^f Surrogate compound.

Source: ESE.

Table 5-15. Reporting Limit Data For Chlorinated Pesticides, EPA 508

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
BHC,A	0.05
BHC,G (Lindane) ^a	0.05
Heptachlor ^a	0.05
Endosulfan I	0.05
Dieldrin ^a	0.10
Endrin ^a	0.10
DDD	0.10
DDT ^a	0.10
Methoxychlor ^a	0.50
BHC,B	0.05
BHC,D	0.05
Aldrin ^a	0.05
Heptachlor epoxide ^a	0.05
Hexachlorobenzene ^a	0.05
Chlordane, G	0.50
Chlordane, A	0.50
DDE	0.10
Endosulfan II	0.10
Endrin aldehyde	0.10
Endosulfan sulfate	0.10
Endrin ketone	0.10
Propachlor ^a	0.10
Toxaphene	1.0

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Table 5-15. Reporting Limit Data For Chlorinated Pesticides, EPA 508 (Continued, Page 2 of 2)

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
Chlordane, Technical	1.0
Trifluralin*	0.50
Aroclor 1016	0.50
Aroclor 1254	0.50
Aroclor 1260	0.50

- * Matrix spike and QC check sample compound.
- * Compound analysis available upon request.

Source: ESE.

Table 5-16. Analytes, Precision, and Accuracy Data For Screening of Polychlorinated Biphenyls, EPA 508A

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
PCBs, as Decachlorobiphenyl	10	80-120

Reference: Screening For Polychlorinated Biphenyls By Perchlorination and Gas Chromatography, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

^b Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

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Table 5-17. Reporting Limit Data For Screening of Polychlorinated Biphenyls, EPA 508A

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
PCBs, as Decachlorobiphenyl	0.1

Source: ESE.

Table 5-18. Analytes, Precision, and Accuracy Data For Chlorinated Herbicides, EPA 515.1

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (%Recovery)
2,4-D ^a	63	48-214
2,4,5-TP/Silvex + der. ^a	69	42-226
Dalapon ^{a,c}	60	30-170
Picloram ^a	52	44-138
Dinoseb ^{a,c}	72	20-130
Dicamba (banvel) ^a	72	38-232
Pentachlorophenol ^a	72	36-224
2,4,5-T ^a	42	68-166
DCAA ^{c,e}	N/A	30-115
2,4-DB	30	48-126
Dichlorprop ^a	30	46-168
MCPPE ^{c,e,f}	30	50-150
MCPA ^{c,e,f}	30	50-150

Reference: EPA Method 515.1 - Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

- ^a Matrix spike and QC check sample compound.
- ^b Accuracy and precision based on method criteria, unless otherwise noted.
- ^c The QC limits are based on the concentration that can be detected reliably according to the ESE Peoria's analytical experience performing the analyses.
- ^d Compound analysis available upon request.
- ^e Compound not listed in the method.
- ^f Surrogate compound.

Source: ESE.

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Table 5-19. Reporting Limit Data For Chlorinated Herbicides, EPA 515.1

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
2,4-D	2.0
2,4,5-TP/Silvex + der.	1.0
Dalapon	2.0
Picloram	2.0
Dinoseb	2.0
Dicamba (banvel)	1.0
Pentachlorophenol	2.0
2,4-DB	1.0
2,4,5-T	2.0
Dichlorprop	2.0
MCPPE ^{a,f}	200
MCPA ^{a,f}	200

- ^a Matrix spike and QC check sample compound.
- ^e Compound analysis available upon request.
- ^f Compound not listed in method.

Source: ESE.

Table 5-20. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
1,1-Dichloroethane	30	80-120
1,2-Dichloroethane ^a	30	80-120
1,1-Dichloroethene ^a	30	80-120
cis-1,2-Dichloroethene ^a	30	80-120
trans-1,2-Dichloroethene ^a	30	80-120
1,2-Dichloropropane ^a	30	80-120
1,3-Dichloropropane	30	80-120
2,2-Dichloropropane	30	80-120
1,1-Dichloropropene	30	80-120
Ethylbenzene ^a	30	80-120
Hexachlorobutadiene	30	80-120
Isopropylbenzene	30	80-120
cis-1,3-Dichloropropene	30	80-120
trans-1,3-Dichloropropene	30	80-120
Naphthalene	30	80-120
Methylene chloride ^a	30	80-120
Styrene ^a	30	80-120
1,1,1,2-Tetrachloroethane	30	80-120
1,1,2,2-Tetrachloroethane	30	80-120
Tetrachloroethene ^a	30	80-120
Toluene ^a	30	80-120
1,2,3-Trichlorobenzene	30	80-120
1,2,4-Trichlorobenzene ^a	30	80-120

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Table 5-20. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2
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Parameter	Precision (RPD)	Aqueous ^b
		Accuracy (% Recovery)
Benzene ^a	30	80-120
Bromobenzene	30	80-120
Bromochloromethane	30	80-120
Bromodichloromethane	30	80-120
Bromoform	30	80-120
Bromomethane	30	80-120
n-Butylbenzene	30	80-120
sec-Butylbenzene	30	80-120
tert-Butylbenzene	30	80-120
Carbon tetrachloride ^a	30	80-120
Chlorobenzene ^a	30	80-120
Chloroform	30	80-120
Chloromethane	30	80-120
Chloroethane	30	80-120
2-Chlorotoluene	30	80-120
4-Chlorotoluene	30	80-120
Dibromochloromethane	30	80-120
4-Isopropyltoluene	30	80-120
n-Propylbenzene	30	80-120
1,2-Dibromo-3-chloropropane	30	80-120
1,2-Dibromoethane	30	80-120
Dibromomethane	30	80-120
1,2-Dichlorobenzene ^a	30	80-120

Table 5-20. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 524.2
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Parameter	Precision (RPD)	Aqueous ^b
		Accuracy (% Recovery)
1,3-Dichlorobenzene	30	80-120
1,4-Dichlorobenzene ^a	30	80-120
Dichlorodifluoromethane	30	80-120
1,1,1-Trichloroethane ^a	30	80-120
1,1,2-Trichloroethane ^a	30	80-120
Trichloroethene ^a	30	80-120
Trichlorofluoromethane	30	80-120
1,2,3-Trichloropropane	30	80-120
1,2,4-Trimethylbenzene	30	80-120
1,3,5-Trimethylbenzene	30	80-120
Vinyl chloride ^a	30	80-120
Xylenes, total ^a	30	80-120
Dichlorobenzene-D4 ^{c,z}	N/A	50-150
4-Bromofluorobenzene ^{c,z}	N/A	50-150

Reference: EPA Method 524.2 - Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^z Surrogate compound.

Table 5-21. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
1,1-Dichloroethane	1.0
1,2-Dichloroethane ^a	0.5
1,1-Dichloroethene ^a	0.5
cis-1,2-Dichloroethene ^a	1.0
trans-1,2-Dichloroethene ^a	1.0
1,2-Dichloropropane ^a	1.0
1,3-Dichloropropane	1.0
2,2-Dichloropropane	1.0
1,1-Dichloropropene	1.0
Ethylbenzene ^a	1.0
Hexachlorobutadiene	1.0
Isopropylbenzene	1.0
cis-1,3-Dichloropropene	1.0
trans-1,3-Dichloropropene	1.0
Naphthalene	1.0
Methylene chloride ^a	1.0
Styrene ^a	1.0
1,1,1,2-Tetrachloroethane	1.0
1,1,2,2-Tetrachloroethane	1.0
Tetrachloroethene ^a	1.0
Toluene ^a	1.0
1,2,3-Trichlorobenzene	1.0
1,2,4-Trichlorobenzene ^a	1.0

Table 5-21. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2
(Continued, Page 2 of 3)

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
Benzene*	0.5
Bromobenzene	1.0
Bromochloromethane	1.0
Bromodichloromethane	1.0
Bromoform	1.0
Bromomethane	2.0
n-Butylbenzene	1.0
sec-Butylbenzene	1.0
tert-Butylbenzene	1.0
Carbon tetrachloride*	0.5
Chlorobenzene*	1.0
Chloroform	1.0
Chloromethane	2.0
Chloroethane	2.0
2-Chlorotoluene	1.0
4-Chlorotoluene	1.0
Dibromochloromethane	1.0
4-Isopropyltoluene	1.0
n-Propylbenzene	1.0
1,2-Dibromo-3-chloropropane	0.5
1,2-Dibromoethane	0.5
Dibromomethane	1.0
1,2-Dichlorobenzene*	1.0

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Table 5-21. Reporting Limit Data for Volatile Organic Compounds, EPA 524.2
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Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
1,3-Dichlorobenzene	1.0
1,4-Dichlorobenzene*	0.5
Dichlorodifluoromethane	2.0
1,1,1-Trichloroethane*	0.5
1,1,2-Trichloroethane*	1.0
Trichloroethene*	1.0
Trichlorofluoromethane	1.0
1,2,3-Trichloropropane	1.0
1,2,4-Trimethylbenzene	1.0
1,3,5-Trimethylbenzene	1.0
Vinyl chloride	0.5
Xylenes, total	1.0

* Matrix spike and QC check sample compound.

Source: ESE.

Table 5-22. Analytes, Precision, and Accuracy Data For N-Methylcarbamoxyl oximes and N-Methyl Carbamates, EPA 531.1

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (%Recovery)
Aldicarb ^{a,c}	9	56-121
Aldicarb sulfone ^{a,c}	12	68-120
Aldicarb sulfoxide ^{a,c}	15	59-131
Carbaryl (Sevin) ^a	18	80-114
Carbofuran ^{a,c}	15	68-119
3-Hydroxycarbofuran ^a	12	90-114
Methomyl ^a	12	92-118
Oxamyl ^a	12	88-112
Methiocarb ^c	30	96-108
Propoxur ^{c,e,f}	30	70-130

Reference: EPA Method 531.1 - Measurement of n-Methylcarbamoxyl oximes and n-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post Column Derivatization, Methods for the Determination of Organic Compounds in Drinking Water, USEPA, (Revision 3.0), 1989.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision criteria based on method, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^f Compound not listed in the method.

Source: ESE.

Table 5-23. Reporting Limit Data for N-Methyl Carbamoyloximes and N-Methyl Carbamates, EPA 531.1

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
Aldicarb ^a	3.0
Aldicarb sulfone ^a	2.0
Aldicarb sulfoxide ^a	4.0
Carbaryl (Sevin) ^a	10
Carbofuran ^a	40
3-Hydroxycarbofuran ^a	10
Methomyl ^a	10
Oxamyl ^a	10
Methiocarb ^c	10
Propoxur ^{a,f}	10

^a Matrix spike and QC check sample compound.

^c Compound analysis available upon request.

^f Compound not listed in the method.

Source: ESE.

Table 5-24. Analyte, Precision, and Accuracy Data For Glyphosate, EPA 547

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
Glyphosate	30	70-130

Reference: Determination of Glyphosate in Drinking Water by Direct Aqueous-Injection HPLC, Post Column Derivatization, and Fluorescence Detection, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

^b Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

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Table 5-25. Reporting Limit Data For Glyphosate, EPA 547

Parameter	<u>Reporting Limit</u>
	Aqueous ($\mu\text{g/L}$)
Glyphosate	6.0

Source: ESE.

Table 5-26. Analyte, Precision, and Accuracy Data For Diquat, EPA 549

Parameter	Aqueous ^b	
	Precision (RPD)	Accuracy (% Recovery)
Diquat	30	70-130

Reference: Determination of Diquat and Paraquat in Drinking Water by Liquid-Solid Extraction and HPLC with Ultraviolet Detection, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

^b Accuracy and precision based on method criteria, unless otherwise noted.

Source: ESE.

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Table 5-27. Reporting Limit Data For Diquat, EPA 549

Parameter	Reporting Limit
	Aqueous ($\mu\text{g/L}$)
Diquat	0.4

Source: ESE.

Table 5-28. Analytes, Precision, and Accuracy Data For Polycyclic Aromatic Hydrocarbons, EPA 550

Parameter	Aqueous ^c	
	Precision (RPD)	Accuracy (% Recovery)
Naphthalene	33	50-110
Acenaphthylene	22	64-110
Acenaphthene	30	60-110
Fluorene	26	62-110
Phenanthrene	43	39-110
Anthracene	13	51-110
Fluoranthene	88	54-126
Pyrene	20	70-110
Benzo(a)anthracene	32	34-118
Chrysene	13	70-118
Benzo(b)fluoranthene	32	32-143
Benzo(k)fluoranthene	23	66-110
Benzo(a)pyrene ^a	64	46-110
Dibenzo(a,h)anthracene	18	52-110
Benzo(g,h,i)perylene	25	42-120
Indeno(1,2,3-cd)pyrene	12	48-110

Table 5-28. Analytes, Precision, and Accuracy Data For Polycyclic Aromatic Hydrocarbons, EPA 550 (Continued, Page 2 of 2)

Parameter	Aqueous ^c	
	Precision (RPD)	Accuracy (% Recovery)
Triphenylene ^a	N/A	48-140

Reference: EPA Method 550 - Determination of Polycyclic Aromatic Hydrocarbons in Drinking Water by Liquid-Liquid Extraction and HPLC with Coupled Ultraviolet and Fluorescence Detection, Methods for the Determination of Organic Compounds in Drinking Water Supplement I, USEPA, July 1990.

- ^a Matrix spike and QC check sample compound.
- ^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
- ^a Surrogate compound.

Source: ESE.

Table 5-29. Reporting Limit Data For Polycyclic Aromatic Hydrocarbons, EPA 550

Parameter	Reporting Limits
	Aqueous ($\mu\text{g/L}$)
Naphthalene	5.0
Acenaphthylene	5.0
Acenaphthene	5.0
Fluorene	5.0
Phenanthrene	0.05
Anthracene	0.05
Fluoranthene	0.05
Pyrene	0.05
Benzo(a)anthracene	0.05
Chrysene	0.05
Benzo(b)fluoranthene	0.05
Benzo(k)fluoranthene	0.05
Benzo(a)pyrene*	0.05
Dibenzo(a,h)anthracene	0.05
Benzo(g,h,i)perylene	0.05
Indeno(1,2,3-cd)pyrene	0.05

* Matrix spike and QC check sample compound.

Source: ESE.

Table 5-30. Analytes, Precision, and Accuracy Data For Purgeable Halocarbons, EPA 601 and SW 5030/8010

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Bromodichloromethane ^d	20	42-172	30	42-172
Bromoform ^d	20	13-159	30	13-159
Bromomethane ^{c,d}	20	15-144	30	15-144
Carbon tetrachloride ^d	20	43-143	30	43-143
Chlorobenzene ^{a,c,d}	24	71-123	50	38-150
Chloroethane ^d	20	46-137	30	46-137
2-Chloroethylvinyl ether ^d	20	14-186	30	14-186
Chloroform ^d	20	49-133	30	49-133
Chloromethane ^{c,d}	20	15-190	30	15-190
Dibromochloromethane ^{c,d}	20	24-190	30	24-190
1,2-Dichlorobenzene ^d	20	37-154	30	37-154
1,3-Dichlorobenzene ^d	20	50-141	30	50-141
1,4-Dichlorobenzene ^d	20	42-143	30	42-143
1,1-Dichloroethane ^d	20	47-132	30	47-132
1,2-Dichloroethane ^d	20	51-147	30	51-147
1,1-Dichloroethene ^{a,c,d}	38	54-182	30	28-167
trans-1,2-Dichloroethene ^d	20	38-155	30	38-155
1,2-Dichloropropane ^d	20	44-156	30	44-156
cis-1,3-Dichloropropene	20	22-178	30	22-178

Table 5-30. Analytes, Precision, and Accuracy Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010 (Continued, Page 2 of 2)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
trans-1,3-Dichloropropene	20	22-178	30	22-178
Dichlorodifluoromethane ^c	20	70-130	30	70-130
Methylene chloride ^d	20	25-162	30	25-162
1,1,2,2-Tetrachloroethane ^d	20	8-184	30	8-184
Tetrachloroethene ^d	20	26-162	30	26-162
1,1,1-Trichloroethane ^d	20	41-138	30	41-138
1,1,2-Trichloroethane ^d	20	39-136	30	39-136
Trichloroethene ^{a,c,d}	26	71-123	30	35-146
Trichlorofluoromethane ^d	20	21-156	30	21-156
Vinyl chloride ^d	20	28-163	30	28-163
Bromochloromethane ^{c,*}	N/A	63-154	N/A	79-115
2-Bromo-1-chloropropane ^{c,*}	N/A	64-146	N/A	60-114
1,4-Dichlorobutane ^{c,*}	N/A	68-138	N/A	55-105

Reference: EPA Method SW 8010-- Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^d Appendix IX compounds.

^{*} Surrogate compound.

Source: ESE.

Table 5-31. Reporting Limit Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Bromodichloromethane ^d	1.0	1.0
Bromoform ^d	5.0	5.0
Bromomethane ^d	5.0	5.0
Carbon tetrachloride ^d	1.0	1.0
Chlorobenzene ^{a,d}	1.0	1.0
Chloroethane ^d	5.0	5.0
2-Chloroethylvinyl ether ^d	5.0	5.0
Chloroform ^d	1.0	1.0
Chloromethane ^d	5.0	5.0
Dibromochloromethane ^d	1.0	1.0
1,2-Dichlorobenzene ^d	1.0	1.0
1,3-Dichlorobenzene ^d	1.0	1.0
1,4-Dichlorobenzene ^d	1.0	1.0
1,1-Dichloroethane ^d	1.0	1.0
1,2-Dichloroethane ^d	1.0	1.0
1,1-Dichloroethene ^{a,d}	2.0	2.0
trans-1,2-Dichloroethene ^d	1.0	1.0
1,2-Dichloropropane ^d	5.0	5.0
cis-1,3-Dichloropropene	1.0	1.0
trans-1,3-Dichloropropene	1.0	1.0
Methylene chloride ^d	2.0	2.0
1,1,2,2-Tetrachloroethane ^d	1.0	1.0
Dichlorodifluoromethane	5.0	5.0

Table 5-31. Reporting Limit Data for Purgeable Halocarbons, EPA 601 and SW 5030/8010
(Continued, Page 2 of 2)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Tetrachloroethene ^d	1.0	1.0
1,1,1-Trichloroethane ^d	1.0	1.0
1,1,2-Trichloroethane ^d	1.0	1.0
Trichloroethene ^{a,d}	1.0	1.0
Trichlorofluoromethane ^d	5.0	5.0
Vinyl chloride ^d	5.0	5.0

^a Matrix spike and QC check sample compound.

^d Appendix IX compounds.

Source: ESE.

Table 5-32. Analytes, Precision, and Accuracy Data for Purgeable Aromatics, EPA 602 and SW 5030/8020

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Benzene ^{a,c,d}	20	68-129	30	74-130
Chlorobenzene ^d	20	55-135	30	55-135
1,2-Dichlorobenzene ^d	20	37-154	30	37-154
1,3-Dichlorobenzene ^d	20	50-141	30	50-141
1,4-Dichlorobenzene ^d	20	42-143	30	42-143
Ethylbenzene ^d	20	32-160	30	32-160
Toluene ^{a,c,d}	20	65-125	30	41-153
Xylenes, total	20	80-126	30	74-128
MTBE ^{e,f}	20	80-120	30	80-120
Trifluorotoluene ^g	N/A	53-126	N/A	16-130

Reference: EPA Method SW 8020--Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

MTBE = methyl tert-butyl ether.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^d Appendix IX compounds.

^f Compound not listed in the method.

^g Surrogate compound.

Source: ESE.

Table 5-33. Reporting Limit Data for Purgeable Aromatics, EPA 602 and SW 5030/8020

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Benzene ^{a,d}	1.0	1.0
Chlorobenzene ^d	1.0	1.0
1,2-Dichlorobenzene ^d	1.0	1.0
1,3-Dichlorobenzene ^d	1.0	1.0
1,4-Dichlorobenzene ^d	1.0	1.0
Ethylbenzene ^d	1.0	1.0
Toluene ^{a,d}	1.0	1.0
Xylenes, total	1.0	1.0
MTBE ^f	5.0	5.0

^a Matrix spike and QC check sample compound.

^d Appendix IX compounds.

^f Compound not listed in the method.

Source: ESE.

Table 5-34. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Aldrin ^{a,c,d}	30	42-122	50	33-137
BHC,A ^d	30	37-134	50	37-134
BHC,B ^d	30	17-147	50	17-147
BHC,D ^d	30	19-140	50	19-140
BHC, G(lindane) ^{a,c,d}	30	40-145	50	30-134
Chlordane, A ^d	30	45-119	50	45-119
Chlordane, G ^d	30	45-119	50	45-119
DDD, PP ^d	30	31-141	50	31-141
DDE, PP ^d	30	30-145	50	30-145
DDT, PP ^{a,c,d}	30	50-149	50	45-145
Dieldrin ^{a,c,d}	30	53-140	50	44-137
Endosulfan, I ^d	30	45-153	50	45-153
Endosulfan, II ^{c,d}	30	15-190	50	15-190
Endosulfan sulfate ^d	30	26-144	50	26-144
Endrin ^{a,c,d}	30	48-143	50	37-153
Endrin aldehyde ^{c,d}	30	50-160	50	50-160
Endrin ketone ^{c,e,f}	30	50-160	50	50-160
Heptachlor ^{a,c,d}	30	44-140	59	30-148
Heptachlor epoxide ^d	30	37-142	50	37-142

Table 5-34. Analytes, Precision, and Accuracy Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080 (Continued, Page 2 of 2)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Methoxychlor ^{c,d}	30	50-160	50	50-160
Toxaphene ^d	30	41-126	50	41-126
PCB-1016 ^{a,d}	30	50-114	50	50-114
PCB-1221 ^d	30	15-178	50	15-178
PCB-1232 ^{c,d}	30	15-190	50	15-190
PCB-1242 ^d	30	39-150	50	39-150
PCB 1248 ^d	30	38-158	50	38-158
PCB-1254 ^d	30	29-131	50	29-131
PCB-1260 ^{a,d}	30	8-127	50	8-127
Mirex ^{c,e,f}	30	50-160	50	50-160
Trifluralin ^{c,e,f}	30	50-160	50	50-160
Chlorpyrifos ^{c,e,f}	30	50-160	50	50-160
Pendimethalin ^{c,e,f}	30	50-160	50	50-160
Tetrachloro-m-xylene ^{c,g}	N/A	52-127	N/A	39-119
Decachlorobiphenyl ^{c,g}	N/A	47-148	N/A	45-127

Reference: EPA Method SW 8080--Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

- ^a Matrix spike and QC check sample compound.
- ^b Accuracy and precision based on method criteria, unless otherwise noted.
- ^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
- ^d Appendix IX compounds.
- ^e Compound analysis available upon request.
- ^f Compound not listed in the method.
- ^g Surrogate compound.

Table 5-35. Reporting Limit Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Aldrin ^{a,d}	0.05	8.0
BHC, A ^d	0.05	8.0
BHC, B ^d	0.05	8.0
BHC, D ^d	0.05	8.0
BHC, G(lindane) ^{a,d}	0.05	8.0
Chlordane, A ^d	0.50	80
Chlordane, G ^d	0.50	80
DDD, PP ^d	0.10	80
DDE, PP ^d	0.10	16
DDT, PP ^{a,d}	0.10	16
Dieldrin ^{a,d}	0.10	16
Endosulfan, I ^d	0.05	8.0
Endosulfan, II ^d	0.05	8.0
Endosulfan sulfate ^d	0.10	16
Endrin ^{a,d}	0.10	16
Endrin aldehyde ^d	0.10	16
Endrin ketone ^{a,f}	0.10	16
Heptachlor ^{a,d}	0.05	8.0
Heptachlor epoxide ^d	0.05	8.0
Methoxychlor ^d	0.50	80
Toxaphene ^d	1.0	160
Mirex ^{a,f}	0.10	16
Trifluralin ^{a,f}	0.05	8.0
Chlorpyrifos ^{a,f}	0.05	8.0
Pendimethalin ^{a,f}	0.10	16

Table 5-35. Reporting Limit Data for Organochlorine Pesticides and PCBs, EPA 608 and SW 3510/3520/3540/3550/8080 (Continued, Page 2 of 2)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
PCB-1016 ^{a,d}	0.50	80
PCB-1221 ^d	0.50	80
PCB-1232 ^d	0.50	80
PCB-1242 ^d	0.50	80
PCB-1248 ^d	0.50	80
PCB-1254 ^d	1.0	160
PCB-1260 ^{a,d}	1.0	160

^a Matrix spike and QC check sample compound.

^d Appendix IX compounds.

^e Compound analysis available upon request.

^f Compound not listed in method.

Source: ESE.

Table 5-36. Analytes, Precision, and Accuracy Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310

Parameter	Aqueous ^c		Solid ^c	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Acenaphthene ^{a,d}	15	31-134	50	30-124
Acenaphthylene ^d	30	30-139	50	30-139
Anthracene ^d	30	30-126	50	30-126
Benzo(a)anthracene ^d	30	30-135	50	30-135
Benzo(a)pyrene ^d	30	30-128	50	30-128
Benzo(b)fluoranthene ^{a,d}	14	30-150	50	30-150
Benzo(ghi)perylene ^d	30	30-116	50	30-116
Benzo(k)fluoranthene ^d	30	30-154	50	30-154
Chrysene ^{a,d}	16	30-150	50	30-150
Dibenz(a,h)anthracene ^d	30	30-110	50	30-110
Fluoranthene ^d	30	30-123	50	30-123
Fluorene ^d	30	30-142	50	30-142
Indeno(1,2,3-cd)pyrene ^d	30	30-116	50	30-116
Naphthalene ^{a,d}	16	30-150	50	30-150
Phenanthrene ^{a,d}	13	30-150	50	30-150
Pyrene ^{a,d}	16	30-150	50	30-150
Triphenylene ^a	N/A	48-140	N/A	25-133

Reference: EPA Method SW 8310--Test Methods for Evaluating Solid Wastes, EPA-SW-846, September 1986.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^d Appendix IX compounds.

^e Surrogate compound.

Source: ESE.

Table 5-37. Reporting Limit Data for Polynuclear Aromatic Hydrocarbons, EPA 610 and SW 3510/3520/3540/3550/8310

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Acenaphthene ^{a,d}	10	330
Acenaphthylene ^d	10	330
Anthracene ^d	0.1	3.3
Benzo(a)anthracene ^d	0.1	3.3
Benzo(a)pyrene ^d	0.1	3.3
Benzo(b)fluoranthene ^{a,d}	0.1	3.3
Benzo(ghi)perylene ^d	0.1	3.3
Benzo(k)fluoranthene ^d	0.1	3.3
Chrysene ^{a,d}	0.1	3.3
Dibenzo(a,h)anthracene ^d	0.1	3.3
Fluoranthene ^d	0.1	3.3
Fluorene ^d	2.0	70
Indeno(1,2,3-cd)pyrene ^d	0.1	3.3
Naphthalene ^{a,d}	10	330
Phenanthrene ^{a,d}	0.1	3.3
Pyrene ^{a,d}	0.1	3.3

^a Matrix spike and QC check sample compound.

^d Appendix IX compounds.

Source: ESE.

Table 5-38. Analytes, Precision, and Accuracy Data for Chlorinated Herbicides, EPA 615 and SW 3510/3520/3540/3550/8150.

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
2,4-D ^{a,c,d}	50	20-144	50	20-129
2,4-DB	50	84-102	50	84-102
2,4,5-T ^{c,d}	50	67-130	50	67-130
2,4,5-TP/Silvex der. ^{a,c,d}	50	20-150	50	20-161
Dicamba (banvel) ^a	50	26-115	50	18-136
Dalapon ^c	50	42-130	50	42-130
Dichloroprop	50	91-103	50	91-103
Dinoseb ^{c,d}	50	74-130	50	74-130
MCPA	50	86-110	50	86-110
MCPD	50	82-106	50	82-106
Pentachlorophenol ^{c,f}	50	70-130	50	70-130
Picloram ^{c,f}	50	70-130	50	70-130
DCAA ^{c,g}	N/A	30-130	N/A	30-130

Reference: EPA Method SW 8150--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^d Appendix IX compounds.

^f Compound not listed in method.

^g Surrogate compound.

Source: ESE.

Table 5-39. Reporting Limit Data for Chlorinated Herbicides, EPA 615 and SW 3510/3520/3540/3550/8150

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
2,4-D ^{a,d}	2.0	100
2,4-DB	2.0	100
2,4,5-T ^{a,d}	1.0	50
2,4,5-TP/Silvex + der. ^{a,d}	1.0	50
Dicamba (banvel) ^a	1.0	50
Dalapon ^a	2.0	100
Dichloroprop	2.0	100
Dinoseb ^{a,d}	2.0	100
MCPA	400	20000
MCPP	400	20000
Pentachlorophenol ^{a,f}	0.2	10
Picloram ^{a,f}	2.0	100

^a Matrix spike and QC check sample compound.

^d Appendix IX compound.

^f Compound not listed in method.

Source: ESE.

Table 5-40. Analytes, Precision, and Accuracy Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Bromacil (Hyvar) ^{c,e,f}	30	50-150	50	50-150
Butachlor (Butanex) ^{a,c,e,f}	30	50-150	50	50-150
Cyanazine (Bladex) ^{a,c,f}	30	25-188	50	46-190
Chlorpyrifos (Lorsban) ^e	30	50-150	50	50-150
Demeton ^a	30	36-99	50	36-99
Diazinon (Basudin) ^a	30	49-85	50	49-85
Dichlorvos ^a	30	49-95	50	49-95
Disulfoton (Mocap) ^a	30	55-109	50	55-109
Fonofos (Dyfonate) ^{c,e,f}	30	50-150	50	50-150
Fenthion (Baytex) ^a	30	9-128	50	9-128
Azinphos methyl (Guthion) ^a	30	16-129	50	16-129
Malathion (Cythion) ^{c,e}	30	50-150	50	50-150
Metolachlor (Dual or Bicep) ^{a,c,f}	13	81-105	42	34-136
Parathion ethyl ^{c,e}	30	50-150	50	50-150
Parathion methyl ^a	30	80-112	50	80-112
Pendimethalin (Prowl) ^{c,f}	30	50-150	50	50-150
Carbofuran (Furadan) ^{c,f}	30	50-150	50	50-150
De-ethyl atrazine (DEA) ^{c,e,f}	30	50-150	50	50-150
De-isopropyl atrazine (DIA) ^{c,e,f}	30	50-150	50	50-150
Fenchlorphos ^{c,e,f}	30	50-150	50	50-150
Phorate (Thimet) ^a	30	36-89	50	36-89

Table 5-40. Analytes, Precision, and Accuracy Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141 (Continued, Page 2 of 2)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Prometon (Pramitol) ^{c,f}	30	50-150	50	50-150
Propachlor (Ramrod) ^{c,e,f}	30	50-150	50	50-150
Propazine (Primatol P) ^{c,e,f}	30	50-150	50	50-150
Simazine (Princep) ^{a,c,f}	30	50-150	50	50-150
Alachlor (Lasso) ^{a,c,f}	34	62-128	43	43-152
Metribuzin (Sencor) ^{a,c,f}	30	50-150	50	50-150
EPTC(Eptam) ^{a,c,f}	32	58-112	73	67-190
Butylate (Sutan) ^{c,f}	30	50-150	50	50-150
Ethalfluralin (Sonalan) ^{c,e,f}	30	50-150	50	50-150
Trifluralin (Treflan) ^{a,c,f}	30	50-150	50	50-150
Atrazine (AAAtrex) ^{a,c,f}	26	50-150	44	46-157
Terbufos (Counter) ^{c,f}	16	79-111	15	88-118
Ethion ^{c,e}	30	50-150	50	50-150
2-NMX ^{c,e}	N/A	52-115	N/A	50-150

Reference: EPA Method SW 8141-Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^e Compound analysis available upon request.

^f Compound not listed in the method.

^{*} Surrogate compound.

Source: ESE.

Table 5-41. Reporting Limit Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Bromacil (Hyvar) ^{a,e,f}	1.0	160
Butachlor (Butanex) ^{a,e,f}	0.50	80
Cyanazine (Bladex) ^{a,f}	1.0	60
Chlorpyrifos (Lorsban)	0.50	80
Demeton ^e	0.50	80
Diazinon (Basudin) ^e	0.50	80
Dichlorvos ^e	0.50	80
Disulfoton (Mocap) ^e	1.0	160
Fonofos (Dyfonate) ^{a,f}	0.50	80
Fenthion (Baytex) ^e	0.50	80
Azinphos methyl (Guthion) ^e	1.0	160
Malathion (Cythion) ^e	0.50	80
Metolachlor (Dual or Bicep) ^{a,f}	0.50	80
Parathion ethyl ^e	0.50	80
Parathion methyl ^e	0.50	80
Pendimethalin (Prowl) ^f	0.50	80
Carbofuran (Furadan) ^f	1.0	160
De-ethyl atrazine (DEA) ^{a,f}	1.0	160
De-isopropyl atrazine (DIA) ^{a,f}	1.0	160
Fenchlorphos ^{a,f}	1.0	160
Phorate (Thimet) ^e	0.50	80
Prometon (Pramitol) ^f	1.0	160
Propachlor (Ramrod) ^{a,f}	1.0	160
Propazine (Primatol P) ^{a,f}	0.50	80
Simazine (Princep) ^{a,f}	0.50	80
Alachlor (Lasso) ^{a,f}	0.50	80
Metribuzin (Sencor) ^{a,f}	0.50	80
EPTC (Eptam) ^{a,f}	0.50	80
Butylate (Sutan) ^f	1.0	160
Ethalfuralin (Sonalan) ^{a,f}	0.50	80

Table 5-41. Reporting Limit Data for Organophosphorus Pesticides, EPA 614/622 and SW 3510/3520/3540/3550/8141 (Continued, Page 2 of 2)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Trifluralin (Treflan) ^{a,f}	0.50	80
Atrazine (AAtrex) ^{a,f}	0.50	80
Terbufos (Counter) ^{a,f}	0.50	80
Ethion ^a	0.50	80

^a Matrix spike and QC check sample compound.

^e Compound analysis available upon request.

^f Compound not listed in method.

Source: ESE.

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Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Acetone ^{c,d,e}	30	61-128	30	61-128
Benzene ^{a,d,i}	11	76-127	21	66-142
Bromodichloromethane ^d	20	35-155	30	35-155
Bromoform ^d	20	45-169	30	45-169
Bromomethane ^{c,d}	20	30-190	30	30-190
Carbon tetrachloride ^d	20	70-140	30	70-140
Chlorobenzene ^{a,d,i}	13	75-130	21	60-133
Chloroethane ^{c,d}	20	30-190	20	30-190
2-Chloroethylvinyl ether ^c	20	30-190	30	30-190
Chloroform ^d	20	51-138	30	51-138
Chloromethane ^{c,d}	20	30-190	30	30-190
Dibromochloromethane ^d	20	53-149	30	53-149
1,2-Dichlorobenzene ^d	20	18-190	30	18-190
1,3-Dichlorobenzene ^d	20	59-156	30	59-156
1,4-Dichlorobenzene ^d	20	18-190	20	18-190
1,1-Dichloroethane ^d	20	59-155	30	59-155
1,2-Dichloroethane ^d	20	49-155	30	49-155
1,1-Dichloroethene ^{a,d,i}	14	61-145	22	59-172
trans-1,2-Dichloroethene ^d	20	54-156	30	54-156

Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 2 of 4)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
1,2-Dichloropropane ^{c,d}	20	30-190	30	30-190
cis-1,3-Dichloropropene ^{c,d}	20	30-190	30	30-190
trans-1,3-Dichloropropene ^d	20	17-183	30	17-183
Ethyl benzene ^d	20	37-162	20	37-162
Methylene chloride ^{c,d}	20	30-190	30	30-190
Methyl ethyl ketone (MEK) ^{c,d,e}	30	60-108	30	60-108
Methyl isobutyl ketone (MIBK) ^{c,d,e}	30	62-130	30	62-130
Methyl tert butyl ether (MTBE) ^{c,e}	30	30-190	30	30-190
Styrene ^{c,d}	30	74-116	30	74-116
1,1,2,2-Tetrachloroethane ^d	20	46-157	30	46-157
Tetrachloroethene ^d	20	64-148	30	64-148
Toluene ^{a,d,i}	13	76-125	21	59-139
1,1,1-Trichloroethane ^d	20	52-162	30	52-162
1,1,2-Trichloroethane ^d	20	52-150	30	52-150
Trichloroethene ^{a,d,i}	14	71-120	24	62-137
Trichlorofluoromethane ^d	20	17-181	30	17-181
Vinyl chloride ^{c,d}	20	30-190	30	30-190
Xylene, total ^{c,d}	30	58-136	30	58-136
Toluene-D8 ^{a,i}	N/A	88-110	N/A	81-117

Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 3 of 4)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
4-Bromofluorobenzene ^{e,i}	N/A	86-115	N/A	74-121
1,2-Dichloroethane-D4 ^{e,i}	N/A	76-114	N/A	70-121
Acrolein ^{c,d,e}	30	52-109	30	52-109
Acrylonitrile ^{c,d,e}	30	70-115	30	70-115
Carbon disulfide ^{c,d}	30	30-117	30	30-117
Chloroprene ^{c,d,e,f}	30	30-190	30	30-190
3-Chloropropene ^{c,d,e,f}	30	30-190	30	30-190
Dichlorodifluoromethane ^{c,d,e}	30	30-190	30	30-190
trans- 1,4-Dichloro-2-butene ^{c,d,e,f}	20	69-109	63	30-121
Ethyl Methacrylate ^{c,d,e}	30	30-190	30	30-190
2-Hexanone ^{c,d}	30	30-190	30	30-190
n-Hexane ^{c,e}	30	30-190	30	30-190
Iodomethane ^{c,d,e}	30	30-190	30	30-190
Methacrylonitrile ^{c,d,e,f}	30	30-190	30	30-190
cis-1,2-Dichloroethene ^c	30	30-130	30	30-130
2-Butanone ^c	30	30-130	30	30-130
4-Methyl-2-pentanone ^c	30	30-130	30	30-130
Methyl methacrylate ^{c,d,e,f}	30	30-190	30	30-190
Propionitrile ^{c,d,e,f}	30	30-190	30	30-190
1,1,1,2-Tetrachloroethane ^{c,d,e,f}	30	87-125	30	87-125
1,2,3-Trichloropropane ^{c,d,e}	30	76-125	30	76-125

Table 5-42. Analytes, Precision, and Accuracy Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 4 of 4)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Vinyl acetate ^{c,d,e}	30	68-130	30	68-130

Reference: EPA Method SW 8240/8260--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition.

- ^a Matrix spike and QC check sample compound.
- ^b Accuracy and precision based on method criteria, unless otherwise noted.
- ^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
- ^d Appendix IX compounds.
- ^e Compound analysis available upon request.
- ^f Compound not listed in method.
- ^g Surrogate compound.
- ⁱ Criteria adopted from USEPA Contract Laboratory Program Statement of Work, March 1990.

Source: ESE.

Table 5-43. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Acetone ^{d,e}	10	10
Benzene ^{a,d}	5.0	5.0
Bromodichloromethane ^d	5.0	5.0
Bromoform ^d	5.0	5.0
Bromomethane ^d	10	10
Carbon tetrachloride ^d	5.0	5.0
Chlorobenzene ^{a,d}	5.0	5.0
Chloroethane ^d	10	10
2-Chloroethylvinyl ether	50	50
Chloroform ^d	5.0	5.0
Chloromethane ^d	10	10
Dibromochloromethane ^d	5.0	5.0
1,2-Dichlorobenzene ^d	5.0	5.0
1,3-Dichlorobenzene ^d	5.0	5.0
1,4-Dichlorobenzene ^d	5.0	5.0
1,1-Dichloroethane ^d	5.0	5.0
1,2-Dichloroethane ^d	5.0	5.0
1,1-Dichloroethene ^{a,d}	5.0	5.0
trans-1,2-Dichloroethene ^d	5.0	5.0
1,2-Dichloropropane ^d	5.0	5.0
cis-1,3-Dichloropropene ^d	5.0	5.0
trans-1,3-Dichloropropene ^d	5.0	5.0
Ethyl benzene ^d	5.0	5.0
Methylene chloride ^d	5.0	5.0
Methyl ethyl ketone ^{d,e}	10	10
Methyl isobutyl ketone ^{d,e}	10	10
Methyl tert butyl ether ^e	10	10
Styrene ^d	5.0	5.0

Table 5-43. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW 5030/8240/8260 (Continued, Page 2 of 3)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
1,1,2,2-Tetrachloroethane ^d	5.0	5.0
Tetrachloroethene ^d	5.0	5.0
Toluene ^{a,d}	5.0	5.0
1,1,1-Trichloroethane ^d	5.0	5.0
1,1,2-Trichloroethane ^d	5.0	5.0
Trichloroethene ^{a,d}	5.0	5.0
Trichlorofluoromethane ^d	10	10
Vinyl chloride ^d	10	10
Xylene, total ^d	5.0	5.0
Acrolein ^{d,e}	50	50
Acrylonitrile ^{d,e}	50	50
Carbon disulfide ^d	5.0	5.0
Chloroprene ^{d,e,f}	5.0	5.0
3-Chloropropene ^{d,e,f}	5.0	5.0
Dichlorodifluoromethane ^{d,e}	10	10
trans-1,4-Dichloro-2-butene ^{d,e,f}	5.0	5.0
Ethyl methacrylate ^{d,e}	5.0	5.0
2-Hexanone ^d	10	10
n-Hexane ^e	10	10
Iodomethane ^{d,e}	5.0	5.0
Methacrylonitrile ^{d,e,f}	5.0	5.0
Methyl methacrylate ^{d,e,f}	5.0	5.0
Propionitrile ^{d,e,f}	5.0	5.0
1,1,1,2-Tetrachloroethane ^{d,e,f}	5.0	5.0
1,2,3-Trichloropropane ^{d,e}	5.0	5.0
Vinyl Acetate ^{d,e}	10	10

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Table 5-43. Reporting Limit Data for Volatile Organic Compounds, EPA 624 and SW
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Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
cis-1,2-Dichloroethene	5.0	5.0
2-Butanone	5.0	5.0
4-Methyl-2-pentanone	5.0	5.0

- ^a Matrix spike and QC check sample compound.
- ^d Appendix IX compounds.
- ^e Compound analysis available upon request.
- ^f Compound not listed in method.

Source: ESE.

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Acenaphthene ^{a,d,i}	31	46-118	19	31-137
Acenaphthylene	30	33-145	50	33-145
Anthracene ^d	30	27-133	50	27-133
1,3-Benzenediol ^{c,e}	30	30-150	50	30-150
Benzidine	30	42-166	50	42-166
Benzo(a)anthracene ^d	30	33-143	50	33-143
Benzo(b)fluoranthene ^d	30	24-159	50	24-159
Benzo(k)fluoranthene ^d	30	11-162	50	11-162
Benzo(a)pyrene ^d	30	17-163	50	17-163
Benzo(ghi)perylene ^{c,d}	30	30-150	50	30-150
Benzyl alcohol ^{c,d}	30	30-150	50	30-150
Butylbenzylphthalate ^{c,d}	30	30-150	50	30-150
bis(2-Chloroethyl)ether ^d	30	12-158	50	12-158
bis(2-Chloroethoxy)-methane ^d	30	33-184	50	33-184
bis(2-Ethylhexyl)-phthalate ^{c,d}	30	30-158	50	30-158
bis(2-Chloroisopropyl)-ether ^d	30	36-166	50	36-166
4-Bromophenylphenyl-ether ^d	30	53-127	50	53-127
Carbazole ^{c,e}	30	30-150	50	30-150
2-Chloronaphthalene ^d	30	60-118	50	60-118
2-Chlorophenol ^{a,d,i}	40	27-123	50	25-102

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Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 2 of 7)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
4-Chloro-3-methylphenol ^{a,d,i}	42	23-97	33	26-103
4-Chlorophenylphenyl ether ^d	30	25-158	50	25-158
Chrysene ^d	30	17-168	50	17-168
Dibenzo(a,h)anthracene ^{c,d}	30	30-150	50	30-150
Di-n-butylphthalate ^{c,d}	30	30-118	50	30-118
1,3-Dichlorobenzene ^{c,d}	30	30-150	50	30-150
1,2-Dichlorobenzene ^d	30	32-129	50	32-129
1,4-Dichlorobenzene ^{a,d,i}	28	36-97	27	28-104
3,3'-Dichlorobenzidine ^{c,d}	30	30-150	50	30-150
2,4-Dichlorophenol ^d	30	39-135	50	39-135
Diethylphthalate ^{c,d}	30	30-150	50	30-150
2,4-Dimethylphenol ^d	30	32-119	50	32-119
Dimethylphthalate ^{c,d}	30	30-150	50	30-150
2,4-Dinitrophenol ^{c,d}	30	30-150	50	30-150
2,4-Dinitrotoluene ^{a,d,i}	38	24-96	47	28-89
2,6-Dinitrotoluene ^d	30	50-158	50	50-158
Di-n-octylphthalate ^{c,d}	30	30-146	50	30-146
Fluoranthene ^d	30	26-137	50	26-137
Fluorene ^d	30	59-121	50	59-121
Hexachlorobenzene ^{c,d}	30	30-150	50	30-150
Hexachlorobutadiene ^d	30	24-116	50	24-116
Hexachlorocyclopentadiene ^{c,d}	30	50-130	50	50-130

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 3 of 7)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Hexachloroethane ^d	30	40-113	50	40-113
Indeno(1,2,3-cd)pyrene ^{c,d}	30	30-150	50	30-150
Isophorone ^d	30	21-196	50	21-196
2-Methyl-4,6-dinitrophenol ^{c,d}	30	30-150	50	30-150
Naphthalene ^d	30	21-133	50	21-133
Nitrobenzene ^d	30	35-180	50	35-180
2-Nitrophenol ^d	30	29-182	50	29-182
4-Nitrophenol ^{a,d,i}	50	10-80	50	11-114
n-Nitrosodimethylamine ^d	30	52-191	50	32-191
n-Nitrosodi-n-propylamine ^{a,d,i}	38	41-116	38	41-126
n-Nitrosodiphenylamine ^d	30	40-112	50	40-112
Pentachlorophenol ^{a,d,i}	50	9-103	47	17-109
Phenanthrene ^d	30	54-120	50	54-120
Phenol ^{a,d,i}	42	12-110	35	26-90
Pyrene ^{a,d,i}	31	26-127	36	35-142
1,2,4-Trichlorobenzene ^{a,d,i}	28	39-98	23	38-107
2,4,6-Trichlorophenol ^d	30	37-144	50	37-144
Acetophenone ^{c,d,e}	30	10-150	50	10-150
2-Acetylaminofluorene ^{c,d,e,f}	30	10-150	50	10-150
4-Aminobiphenyl ^{c,d,e}	30	10-150	50	10-150
Aniline ^{c,d,e}	30	10-150	50	10-150

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Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 4 of 7)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Aramite ^{c,d,e,f}	30	10-150	50	10-150
1,4-Benzenediamine ^{c,d,e,f}	30	10-150	50	10-150
p-Benzoquinone ^{c,d,e,f}	30	10-150	50	10-150
4-Chloroaniline ^{c,d,e}	30	10-150	50	10-150
Chlorobenzilate ^{c,d,e,f}	30	10-150	50	10-150
1-Chloronaphthalene ^{c,d,e}	30	10-150	50	10-150
Dibenz(a,j)acridine ^{c,d,e}	30	10-150	50	10-150
Diallate ^{c,d,e,f}	30	10-150	50	10-150
Dibenzofuran ^{c,d,e}	30	10-150	50	10-150
2,6-Dichlorophenol ^{c,d,e}	30	10-150	50	10-150
Dimethoate ^{c,d,e,f}	30	10-150	50	10-150
p-(Dimethylamino)azo-benzene ^{c,d,e}	30	10-150	50	10-150
7,12-Dimethylbenz(a)anthracene ^{c,d,e}	30	10-150	50	10-150
3,3-Dimethylbenzidine ^{c,d,e,f}	30	10-150	50	10-150
m-Dinitrobenzene ^{c,d,e,f}	30	10-150	50	10-150
Diphenylamine ^{c,d,e}	30	10-150	50	10-150
1,2-Diphenylhydrazine ^{c,d,e}	30	10-150	50	10-150
Ethylmethanesulfonate ^{c,d,e}	30	10-150	50	10-150
a,a-Dimethylphenylamine ^{c,d,e}	30	10-150	50	10-150
Hexachlorophene ^{c,d,e,f}	30	10-150	50	10-150
Hexachloropropene ^{c,d,e,f}	30	10-150	50	10-150

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 5 of 7)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (%Recovery)	Precision (RPD)	Accuracy (%Recovery)
Isosafrole ^{c,d,e,f}	30	10-150	50	10-150
Methapyrilene ^{c,d,e,f}	30	10-150	50	10-150
3-Methylcholanthrene ^{c,d,e}	30	10-150	50	10-150
Methylmethanesulfonate ^{c,d,e,f}	30	10-150	50	10-150
2-Methylnaphthalene ^{c,d,e}	30	10-150	50	10-150
2-Methylphenol(o-Cresol) ^{c,d,e}	30	10-150	50	10-150
3-Methylphenol(m-Cresol) ^{c,d,e,f}	30	10-150	50	10-150
4-Methylphenol(p-Cresol) ^{c,d,e}	30	10-150	50	10-150
1-Naphthylamine ^{c,d,e}	30	10-150	50	10-150
2-Naphthylamine ^{c,d,e}	30	10-150	50	10-150
2-Nitroaniline ^{c,d,e}	30	10-150	50	10-150
3-Nitroaniline ^{c,d,e}	30	10-150	50	10-150
4-Nitroaniline ^{c,d,e}	30	10-150	50	10-150
N-Nitrosodiethylamine ^{c,d,e,f}	30	10-150	50	10-150
N-Nitroso-di-n-butylamine ^{c,d,e}	30	10-150	50	10-150
N-Nitrosomethyl-ethylamine ^{c,d,e,f}	30	10-150	50	10-150
N-Nitrosomorpholine ^{c,d,e,f}	30	10-150	50	10-150
N-Nitrosopiperidine ^{c,d,e}	30	10-150	50	10-150
4-Nitroquinoline-1-oxide ^{c,d,e,f}	30	10-150	50	10-150
N-Nitrosopyrrolidine ^{c,d,e,f}	30	10-150	50	10-150
1,4-Naphthoquinone ^{c,d,e,f}	30	10-150	50	10-150

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Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 6 of 7)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
5-Nitro-o-toluidine ^{c,d,e,f}	30	10-150	50	10-150
Pentachlorobenzene ^{c,d,e}	30	10-150	50	10-150
Pentachloronitrobenzene ^{c,d,e}	30	10-150	50	10-150
Phenacetin ^{c,d,e}	30	10-150	50	10-150
Phorate ^{c,d,e}	30	10-150	50	10-150
Parathion ^{c,d,e}	30	10-150	50	10-150
2-Picoline ^{c,d,e}	30	10-150	50	10-150
Pronamide ^{c,d,e}	30	10-150	50	10-150
Pyridine ^{c,d,e,f}	30	10-150	50	10-150
1,2,4,5-Tetrachloro- benzene ^{c,d,e}	30	10-150	50	10-150
2,3,4,6-Tetrachlorophenol ^{c,d,e}	30	10-150	50	10-150
2,4,5-Trichlorophenol ^{c,d,e}	30	10-150	50	10-150
1,3,5-Trinitrobenzene ^{c,d,e,f}	30	10-150	50	10-150
o-Toluidine ^{c,d,e,f}	30	10-150	50	10-150
Safrole ^{c,d,e,f}	30	10-150	50	10-150
Nitrobenzene-D5 ^{c,e}	N/A	35-114	N/A	23-120
2-Fluorobiphenyl ^{c,e}	N/A	43-116	N/A	30-115
p-Terphenyl-D4 ^{c,e}	N/A	33-141	N/A	18-137
Phenol-D6 ^{c,e}	N/A	10-110	N/A	24-113
2-Fluorophenol ^{c,e}	N/A	21-110	N/A	25-121
2,4,6-Tribromophenol ^{c,e}	N/A	10-123	N/A	19-122

Table 5-44. Analytes, Precision, and Accuracy Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 7 of 7)

Parameter	Aqueous ^b		Solid ^b	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
2-Chlorophenol-D4 ^{c,z}	N/A	33-110	N/A	20-130
1,2-Dichlorobenzene-D4 ^{c,z}	N/A	16-110	N/A	20-130
Benzoic acid ^{c,e}	30	10-150	50	10-150
Dinoseb ^{c,d,e}	30	10-150	50	10-150
Disulfoton ^{c,d,e}	30	10-150	50	10-150
Famphur ^{c,d,e}	30	10-150	50	10-150
Isodrin ^{c,d,e}	30	10-150	50	10-150
Kepone ^{c,d,e}	30	10-150	50	10-150
Methyl parathion ^{c,d,e}	30	10-150	50	10-150
Tetraethyl dithiopyrophosphate (Sulfotepp) ^{c,d,e}	30	10-150	50	10-150
Thionazine ^{c,e}	30	10-150	50	10-150
O,O,O-Triethyl phosphorothioate ^{c,d,e}	30	10-150	50	10-150

Reference: EPA Method SW 8270--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

- ^a Matrix spike and QC check sample compound.
- ^b Accuracy and precision based on method criteria, unless otherwise noted.
- ^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.
- ^d Appendix IX compounds.
- ^e Compound analysis available upon request.
- ^f Compound not listed in method.
- ^z Surrogate compound.
- ⁱ Criteria adopted from USEPA Contract Laboratory Program Statement of Work, March 1990.

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Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW
3510/3520/3540/3550/8270

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Acenaphthene ^{a,d}	10	330
Acenaphthylene	10	330
Anthracene ^d	10	330
1,3-Benzenediol ^a	10	330
Benzidine	50	1600
Benzo(a)anthracene ^d	10	330
Benzo(b)fluoranthene ^d	10	330
Benzo(k)fluoranthene ^d	10	330
Benzo(a)pyrene ^d	10	330
Benzo(ghi)perylene ^d	10	330
Benzyl alcohol ^d	10	330
Butylbenzylphthalate ^d	10	330
bis(2-Chloroethyl)ether ^d	10	330
bis(2-Chloroethoxy)methane ^d	10	330
bis(2-Ethylhexyl)phthalate ^d	10	330
bis(2-Chloroisopropyl)ether ^d	10	330
4-Bromophenylphenyl-ether ^d	10	330
Carbazole ^a	10	330
2-Chloronaphthalene ^d	10	330
2-Chlorophenol ^{a,d}	10	330
4-Chloro-3-methylphenol ^{a,d}	10	330
4-Chlorophenylphenyl ether ^d	10	330
Chrysene ^d	10	330
Dibenzo(a,h)anthracene ^d	10	330
Di-n-butylphthalate ^d	10	330
1,3-Dichlorobenzene ^d	10	330
1,2-Dichlorobenzene ^d	10	330
1,4-Dichlorobenzene ^{a,d}	10	330
3,3'-Dichlorobenzidine ^d	20	660

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 2 of 6)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
2,4-Dichlorophenol ^d	10	330
Diethylphthalate ^d	10	330
2,4-Dimethylphenol ^d	10	330
Dimethylphthalate ^d	10	330
2,4-Dinitrophenol ^d	50	1600
2,4-Dinitrotoluene ^{a,d}	10	330
2,6-Dinitrotoluene ^d	10	330
Di-n-octylphthalate ^d	10	330
Fluoranthene ^d	10	330
Fluorene ^d	10	330
Hexachlorobenzene ^d	10	330
Hexachlorobutadiene ^d	10	330
Hexachlorocyclopentadiene ^d	10	330
Hexachloroethane ^d	10	330
Indeno(1,2,3-cd)pyrene ^d	10	330
Isophorone ^d	10	330
2-Methyl-4,6-dinitrophenol ^d	50	1600
Naphthalene ^d	10	330
Nitrobenzene ^d	10	330
2-Nitrophenol ^d	10	330
4-Nitrophenol ^{a,d}	50	1600
n-Nitrosodimethylamine ^d	10	330
n-Nitrosodi-n-propylamine ^{a,d}	10	330
n-Nitrosodiphenylamine ^d	10	330
Pentachlorophenol ^{a,d}	50	1600
Phenanthrene ^d	10	330
Phenol ^{a,d}	10	330
Pyrene ^{a,d}	10	330
1,2,4-Trichlorobenzene ^{a,d}	10	330

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Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 3 of 6)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
2,4,6-Trichlorophenol ^d	10	330
Acetophenone ^{d,e}	10	330
2-Acetylaminofluorene ^{d,e,f}	10	330
4-Aminobiphenyl ^{d,e}	10	330
Aniline ^{d,e}	10	330
Aramite ^{d,e,f}	10	330
1,4-Benzenediamine ^{d,e,f}	10	330
p-Benzoquinone ^{d,e,f}	10	330
4-Chloroaniline ^{d,e}	10	330
Chlorobenzilate ^{d,e,f}	10	330
1-Chloronaphthalene ^{d,e}	10	330
Dibenz(a,j)acridine ^{d,e}	10	330
Diallate ^{d,e,f}	10	330
Dibenzofuran ^{d,e}	10	330
2,6-Dichlorophenol ^{d,e}	10	330
Dimethoate ^{d,e,f}	10	330
p-(Dimethylamino)azobenzene ^{d,e}	10	330
7,12-Dimethylbenz(a)anthracene ^{d,e}	10	330
3,3-Dimethylbenzidine ^{d,e,f}	10	330
m-Dinitrobenzene ^{d,e,f}	10	330
Diphenylamine ^{d,e}	10	330
1,2-Diphenylhydrazine ^{d,e}	10	330
Ethylmethanesulfonate ^{d,e}	10	330
a,a-Dimethylphenethylamine ^{d,e}	10	330
Hexachlorophene ^{d,e,f}	10	330
Hexachloropropene ^{d,e,f}	10	330
Isosafrole ^{d,e,f}	10	330
Methapyrilene ^{d,e,f}	10	330
3-Methylcholanthrene ^{d,e}	10	330

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 4 of 6)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Methylmethanesulfonate ^{d,e,f}	10	330
2-Methylnaphthalene ^{d,e}	10	330
2-Methylphenol (o-Cresol) ^{d,e}	10	330
3-Methylphenol (m-Cresol) ^{d,e,f}	10	330
4-Methylphenol (p-Cresol) ^{d,e}	10	330
1-Naphthylamine ^{d,e}	10	330
2-Naphthylamine ^{d,e}	10	330
2-Nitroaniline ^{d,e}	50	1600
3-Nitroaniline ^{d,e}	50	1600
4-Nitroaniline ^{d,e}	50	1600
N-Nitrosodiethylamine ^{d,e,f}	10	330
N-Nitroso-di-n-butylamine ^{d,e}	10	330
N-Nitrosomethylethylamine ^{d,e,f}	10	330
N-Nitrosomorpholine ^{d,e,f}	10	330
N-Nitrosopiperidine ^{d,e}	10	330
4-Nitroquinoline-1-oxide ^{d,e,f}	10	330
N-Nitrosopyrrolidine ^{d,e,f}	10	330
1,4-Naphthoquinone ^{d,e,f}	10	330
5-Nitro-o-toluidine ^{d,e,f}	10	330
Pentachlorobenzene ^{d,e}	10	330
Pentachloronitrobenzene ^{d,e}	10	330
Phenacetin ^{d,e}	10	330

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Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 5 of 6)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Phorate ^{d,e}	10	330
Parathion ^{d,e}	10	330
2-Picoline ^{d,e}	10	330
Pronamide ^{d,e}	10	330
Pyridine ^{d,e,f}	10	330
1,2,4,5-Tetrachlorobenzene ^{d,e}	10	330
2,4,5-Trichlorophenol ^{d,e}	50	1600
2,3,4,6-Tetrachlorophenol ^{d,e}	10	330
1,3,5-Trinitrobenzene ^{d,e,f}	10	330
o-Toluidine ^{d,e,f}	10	330
Safrole ^{d,e,f}	10	330
Benzoic acid ^e	10	330
Dinoseb ^{d,e}	10	330
Disulfoton ^{d,e}	10	330
Famphur ^{d,e}	10	330
Isodrin ^{d,e}	10	330
Kepone ^{d,e}	10	330
Methyl parathion ^{d,e}	10	330
Tetraethyl dithiopyrophosphate (Sulfotepp) ^{d,e}	10	330

Table 5-45. Reporting Limit Data for Semivolatile Organic Compounds, EPA 625 and SW 3510/3520/3540/3550/8270 (Continued, Page 6 of 6)

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Thionazine ^a	10	330
O,O,O-Triethyl phosphorothioate ^{d,e}	10	330

- ^a Matrix spike and QC check sample compound.
- ^d Appendix IX compounds.
- ^e Compound analysis available upon request.
- ^f Compound not listed in method.

Source: ESE.

Table 5-46. Analytes, Precision, and Accuracy Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography (HPLC), SW 8330

Parameter	Aqueous ^c		Solid ^c	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
HMX	13	84-111	18	80-116
RDX ^a	30	51-111	18	71-107
1,3,5-Trinitrobenzene ^a	28	46-102	25	65-115
1,3-Dinitrobenzene	37	58-132	30	70-130
Methyl-2,4,6-Trinitro-phenylnitramine (Tetryl)	21	67-109	46	65-157
Nitrobenzene ^a	32	44-108	24	72-120
2,4,6-Trinitrotoluene ^a	38	48-124	23	72-118
2,4-Dinitrotoluene ^a	21	60-102	19	68-106
2,6-Dinitrotoluene	26	67-119	44	58-146
o-Nitrotoluene	28	53-109	22	70-114
m-Nitrotoluene	48	40-136	48	40-136
p-Nitrotoluene	26	60-112	26	60-112
4-Amino-2,6-dinitrotoluene	30	70-130	30	70-130
2-Amino-4,6-dinitrotoluene	30	70-130	30	70-130
3,4-Dinitrotoluene ^{c,*}	N/A	30-150	N/A	30-150

Reference: EPA Method SW 8330--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

^a Matrix spike and QC check sample compound.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^{*} Surrogate compound.

Table 5-47. Reporting Limit Data for Nitroaromatics and Nitroamines by High Performance Liquid Chromatography, SW 8330

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
HMX	0.65	650
RDX ^a	0.30	300
1,3,5-Trinitrobenzene ^a	0.25	250
1,3-Dinitrobenzene	0.15	150
Methyl-2,4,6-Trinitro- phenylnitramine	0.20	200
Nitrobenzene ^a	0.40	400
2,4,6-Trinitrotoluene ^a	0.20	200
2,4-Dinitrotoluene ^a	0.15	150
2,6-Dinitrotoluene	0.25	250
o-Nitrotoluene	0.7	700
m-Nitrotoluene	1.0	700
p-Nitrotoluene	0.7	1,000
4-Amino-2,6-dinitrotoluene	0.7	1,000
2-Amino-4,6-dinitrotoluene	0.7	1,000

^a Matrix spike and QC check sample compound.

Source: ESE.

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Table 5-48. Analytes, Precision, and Accuracy Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, California Method Modified

Parameter	Aqueous ^c		Solid ^c	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Diesel ^a	30	50-150	50	50-150
Gasoline	30	50-150	50	50-150
Jet Fuel	30	50-150	50	50-150
Unidentified Compound	30	50-150	50	50-150
Mineral Spirits	30	50-150	50	50-150
Motor Oil	30	50-150	50	50-150

Reference: California Method Revision 2.0, August 1991, Southern California Laboratory Hazardous Materials Unit.

^a Matrix spike and QC check sample compound.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-49. Reporting Limit Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, California Method Modified

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Diesel*	0.5	10
Gasoline	0.5	10
Jet Fuel	0.5	10
Unidentified Compound	0.5	10
Mineral Spirits	0.5	10
Motor Oil	10	160

* Matrix spike and QC check sample compound.

Source: ESE.

Table 5-50. Analytes, Precision, and Accuracy Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, SW 5030/8015 Modified

Parameter **	Aqueous ^c		Solid ^c	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Methanol ^f	30	50-150	50	50-150
Ethanol	30	50-150	50	50-150
Isopropanol ^f	30	50-150	50	50-150
N-Propanol ^f	30	50-150	50	50-150
N-Butanol ^f	30	50-150	50	50-150
T-Butanol ^f	30	50-150	50	50-150
Isobutanol ^{d,f}	30	50-150	50	50-150
Isoamyl alcohol ^f	30	50-150	50	50-150
Acetaldehyde ^f	30	50-150	50	50-150
Ethyl ether	30	50-150	50	50-150
Ethyl acetate ^f	30	50-150	50	50-150
1,2-Epoxybutane ^f	30	50-150	50	50-150
2-Methoxyethanol ^f	30	50-150	50	50-150
2-Ethoxyethanol ^f	30	50-150	50	50-150
2-Butoxyethanol ^f	30	50-150	50	50-150
Methyl ethyl ketone (MEK)	30	50-150	50	50-150
1,4-Dioxane ^{d,f}	30	50-150	50	50-150
Isopropyl acetate ^f	30	50-150	50	50-150
Cyclohexanone ^f	30	50-150	50	50-150
Ethylene glycol ^f	30	50-150	50	50-150
Diethylene glycol ^f	30	50-150	50	50-150
Pentachlorethane ^{d,f}	30	50-150	50	50-150
Acetonitrile ^{d,f}	30	50-150	50	50-150

Reference: EPA Method SW 8015--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^d Appendix IX compounds.

^f Compound not listed in the method.

** The target requested is spiked.

Table 5-51. Reporting Limit Data for Nonhalogenated Volatile Organics by Flame Ionization Detector, SW 5030/8015 Modified

Parameter	Reporting Limits	
	Aqueous (mg/L)	Solid (mg/kg)
Methanol ^f	5.0	5.0
Ethanol	5.0	5.0
Isopropanol ^f	5.0	5.0
N-Propanol ^f	5.0	5.0
N-Butanol ^f	5.0	5.0
T-Butanol ^f	5.0	5.0
Isobutanol ^{d,f}	5.0	5.0
Isoamyl alcohol ^f	5.0	5.0
Acetaldehyde ^f	5.0	5.0
Ethyl ether	5.0	5.0
Ethyl acetate ^f	10	10
1,2-Epoxybutane ^f	5.0	5.0
2-Methoxyethanol ^f	5.0	5.0
2-Ethoxyethanol ^f	5.0	5.0
2-Butoxyethanol ^f	5.0	5.0
Methyl ethyl ketone (MEK)	5.0	5.0
1,4-Dioxane ^{d,f}	5.0	5.0
Isopropyl acetate ^f	10	10
Cyclohexanone ^f	5.0	5.0
Ethylene glycol ^f	100	100
Diethylene glycol ^f	100	100
Pentachlorethane ^{d,f}	5.0	5.0
Acetonitrile ^{d,f}	5.0	5.0

^d Appendix IX compound.

^f Compound not listed in method.

Source: ESE.

Table 5-52. Analytes, Precision, and Accuracy Data for Polynuclear Aromatic Hydrocarbons by Flame Ionization Detector, SW 3510/3520/3540/3550/8100

Parameter ^{a*}	Aqueous ^c		Solid ^c	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
Acenaphthene	30	30-150	50	30-150
Acenaphthylene	30	30-150	50	30-150
Anthracene	30	30-150	50	30-150
Benzo(a)anthracene	30	30-150	50	30-150
Benzo(a)pyrene	30	30-150	50	30-150
Benzo(b)fluoranthene	30	30-150	50	30-150
Benzo(k)fluoranthene	30	30-150	50	30-150
Benzo(ghi)perylene	30	30-150	50	30-150
Chrysene	30	30-150	50	30-150
Dibenzo(a,h)anthracene	30	30-150	50	30-150
Fluoranthene	30	30-150	50	30-150
Fluorene	30	30-150	50	30-150
Indeno(1,2,3-cd)pyrene	30	30-150	50	30-150
Naphthalene	30	30-150	50	30-150
Phenanthrene	30	30-150	50	30-150
Pyrene	30	30-150	50	30-150

Reference: EPA Method SW 8100--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

^{a*} The target requested is spiked.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

Source: ESE.

Table 5-53. Reporting Limit Data for Polynuclear Aromatic Hydrocarbons by Flame Ionization Detector, SW 3510/3520/3540/3550/8100

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
Acenaphthene	10	330
Acenaphthylene	10	330
Anthracene	10	330
Benzo(a)anthracene	10	330
Benzo(a)pyrene	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(ghi)perylene	10	330
Chrysene	10	330
Dibenzo(a,h)anthracene	10	330
Fluoranthene	10	330
Fluorene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Naphthalene	10	330
Phenanthrene	10	330
Pyrene	10	330

Source: ESE.

Table 5-54. Analytes, Precision, and Accuracy Data for Phenols, SW 3510/3520/3540/3550/8040

Parameter	Aqueous ^c		Solid ^c	
	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
2-sec-butyl-4,6-Dinitrophenol (DNBP)	30	30-150	50	30-150
4-Chloro-3-methylphenol ^a	30	30-150	50	30-150
2-Chlorophenol ^{a,b}	30	38-126	50	30-150
Cresols (methyl phenols)	30	30-150	50	30-150
2-Cyclohexyl-4,6-dinitrophenol	30	30-150	50	30-150
2,4-Dichlorophenol ^b	30	44-119	50	30-150
2,6-Dichlorophenol	30	30-150	50	30-150
2,4-Dimethylphenol ^b	30	24-118	50	30-150
2,4-Dinitrophenol ^b	30	12-145	50	30-150
2-Methyl-4,6-dinitrophenol ^b	30	30-136	50	30-150
2-Nitrophenol ^b	30	43-117	50	30-150
4-Nitrophenol ^{a,b}	30	13-110	50	30-150
Pentachlorophenol ^{a,b}	30	36-134	50	30-150
Phenol ^{a,b}	30	23-108	50	30-150
Tetrachlorophenols	30	30-150	50	30-150
Trichlorophenols	30	30-150	50	30-150
2,4,6-Trichlorophenol ^{a,b}	30	53-119	50	30-150
2-Fluorophenol ^a	N/A	30-150	N/A	30-150
2,4,6-Tribromophenol ^a	N/A	30-150	N/A	30-150

Reference: EPA Method SW 8040--Test Methods for Evaluating Solid Wastes, EPA-SW-846 3rd Edition, September 1986.

^a Matrix spike and QC check sample compound.

^b Accuracy and precision data based on method criteria, unless otherwise noted.

^c The QC limits are based on the concentration that can be detected reliably according to ESE Peoria's analytical experience performing the analyses.

^a Surrogate compound.

Source: ESE.

Table 5-55. Reporting Limit Data for Phenols, SW 3510/3520/3540/3550/8040

Parameter	Reporting Limits	
	Aqueous ($\mu\text{g/L}$)	Solid ($\mu\text{g/kg}$)
2-sec-butyl-4,6-Dinitrophenol (DNBP)	5.0	5.0
4-Chloro-3-methylphenol ^a	10	10
2-Chlorophenol ^a	5.0	5.0
Cresols (methyl phenols)	5.0	5.0
2-Cyclohexyl-4,6-dinitrophenol	5.0	5.0
2,4-Dichlorophenol	5.0	5.0
2,6-Dichlorophenol	5.0	5.0
2,4-Dimethylphenol	5.0	5.0
2,4-Dinitrophenol	10	10
2-Methyl-4,6-dinitrophenol	5.0	5.0
2-Nitrophenol	5.0	5.0
4-Nitrophenol ^a	5.0	5.0
Pentachlorophenol ^a	5.0	5.0
Phenol ^a	5.0	5.0
Tetrachlorophenols	5.0	5.0
Trichlorophenols	5.0	5.0
2,4,6-Trichlorophenol ^a	5.0	5.0

Source: ESE.

^a Matrix spike and QC check sample compound.

6.0 SAMPLE HANDLING PROCEDURES

6.1 INTRODUCTION

The laboratory is able to provide field teams with sampling kits that contain all the required sampling bottles, documents, labels, and preservative solutions as needed for any field sampling effort. Requirements for any field sampling performed by the ESE Peoria Laboratory will be documented in a site specific QAPP. This section of the CQAP details sample handling requirements in the laboratory.

6.2 SAMPLE CONTAINERS CLEANING PROCEDURES

6.2.1 CLEANING PROCEDURES

ESE Peoria uses commercially cleaned sample containers. At a minimum, only Type 200 precleaned sample containers, cleaned according to EPA protocols, and provided with a certificate of cleanliness are used. The certificates are kept on file in the QA/QC office. Table 6-1 summarizes the application of these cleaning procedures. Clean sample containers are stored in a storage shed and a preparation room, both separate from the laboratory.

All sample containers are prepared for shipment in a separate room from the laboratory. Upon receipt of precleaned sample containers, the purchase order form is dated with date of receipt by the laboratory purchasing personnel and the purchase order form is filed. Documentation associated with the sample containers such as lot numbers and certification statements for the containers are maintained and filed in the QA/QC office. Containers are individually labeled or barcoded by the manufacturer referencing lot numbers. It is not necessary to maintain records of lot numbers used for a particular project.

6.2.2 TYPES OF WATER

Deionized (DI) water is defined as ESE water that has been treated by passing it through a standard resin column and an activated carbon unit. The water contains no detectable (i.e., ESE's routine detection limits) heavy metals or inorganic compounds of analytical

Table 6-1. Sample Container Cleaning Procedures*

LEVEL ONE	
Glassware and plasticware receive full EPA quality assurance treatment. Containers are cleaned according to EPA recommended wash procedures and undergo strict quality control analysis. Additional sampling custody seals for bottle closures are included in each case. Each case of containers is then custody sealed - chain-of-custody is intact right from the start. Each container is lot number labeled for traceability to the enclosed certificate of analysis.	
CLEANING PROCEDURE A	
1. Bottles, liners, and caps are washed in laboratory-grade, nonphosphate detergent.	
2. Rinsed three times with distilled water.	
3. Rinsed with 1:1 nitric acid.	
4. Rinsed three times with ASTM Type 1 organic-free water.	
5. Oven-dried for one hour.	
6. Rinsed with hexane.	
7. Oven-dried for one hour.	

Note: Cleaning protocols are applied by commercial supplier.

* Provided by Eagle-Picher, 1993, p. 3.

interest and is relatively free of organic compounds. The water is acceptable for use in the initial rinsing of laboratory glassware. Ultrapure water, used for instrumentation, is defined as ESE Milli-Q water that has been additionally treated through a Milli-Q® treatment system and contains no organic compounds of analytical interest above ESE's routine detection limits.

Water, distilled or deionized, other than ESE-treated water may be used if it is of documented equivalent quality. Commercially available distilled water is used for volatile organic method blanks and trip blanks. The water contains no detectable volatile organic compounds of analytical interest. Documentation is maintained to demonstrate reliability and purity of analyte free water sources.

6.3 SAMPLING CONTAINERS, VOLUMES, HOLDING TIMES AND PRESERVATION

6.3.1 CONTAINERS AND SAMPLE HOLDING TIMES

Table 6-2 identifies the proper containers, preservation techniques, and maximum holding times established by the EPA (40 CFR Part 136). The maximum holding times in Table 6-2 apply to water and soils as noted. If maximum holding times are exceeded, the Project Manager notifies the client and the conversation is documented in the Project Manager's telephone record.

Samples that exceed the regulatory holding times will be flagged by the Project Manager or Laboratory Coordinator in the final deliverable. Sample container sizes for water and soil matrices are one liter and 500 mL, respectively, except for VOAs. Sample container sizes for water and soil matrices for VOAs are 44 mL and 60 mL (wide mouth), respectively. (Water samples for VOAs should be collected in duplicate.)

6.3.2 SAMPLE PRESERVATION

Sample preservation is generally performed in the laboratory by means of adding the preservatives to the containers before shipment to the field, unless preservation in the field is requested. Sample containers for volatile analysis (water only) and carbamates are pre-preserved by the manufacturer and are shipped to the field as received from the manufacturer. All preservatives are prepared from reagent grade acids and chemicals.

6.4 SAMPLE SHIPPING FROM THE FIELD TO THE LABORATORY

A typical environmental sample consists of some type of soil or water matrix; however, other types of samples such as tissues or dust wipes are collected. Whatever the field sample type, the field crew must package each sample container to ensure its integrity inside the shipping container. This packaging may include packing materials such as Bubble Wrap® or styrofoam fillers.

Sample containers are typically shipped by bonded courier to the ESE laboratory. Samples are shipped by overnight delivery or as soon as possible after collection (usually daily), with a receiving signature required. Sample receipt and log-in at the Peoria Laboratory is performed by the Sample Custodian, as described in Section 7.3.

If the samples require chilling/freezing, the sample containers are isolated from the chilling/freezing materials using appropriate, waterproof materials such as plastic bags which the laboratory provides in the sampling kits. Typically, wet ice is used to chill the samples; reusable blue ice-type chilling products are not used, unless requested by the client, due to possible chemical interferences. If a sample must be kept frozen in a solid state, dry ice is used.

The Chain-of-Custody forms for the samples in each shipping container are sealed in a plastic Ziploc® bag and taped to the inside of the container. ESE Peoria's policy requires sealing all sample shipping containers with evidence tape prior to shipping.

Samples received by the laboratory that require pH adjustment for preservation are randomly checked to determine that the pH adjustment was made. Sample custodians check the first shipment received each day using unit resolution pH paper. The results are recorded in a logbook. Any problems encountered are reported to the Project Manager or Laboratory Coordinator. Upon client request, additional shipments can be checked for proper preservation techniques.

Table 6-2. Required Containers, Preservation Techniques, and Holding Times

Measurement	Container ¹	Preservation	Maximum Holding Time ² (Waters and Soils)
Metals			
Chromium VI	P	Cool, 4°C	24 hours ³
Mercury	P	HNO ₃ to pH < 2	28 days
Metals, except chromium VI and mercury (filtered and unfiltered)	P	HNO ₃ to pH < 2	6 months
Inorganic Tests			
Acidity	P, G	Cool, 4°C	14 days ³
Alkalinity	P, G	Cool, 4°C	14 days ³
Ammonia	P, G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
BOD	P, G	Cool, 4°C	48 hours ³
Bromide	P, G	Cool, 4°C	28 days ³
BOD, carbonaceous	P, G	Cool, 4°C	48 hours ³
COD	P, G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
Chloride	P, G	Cool, 4°C	28 days ³
Chlorine, total	P, G	Cool, 4°C	Analyze immediately ^{3,7}
Color	P, G	Cool, 4°C	48 hours ³
Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH > 12, 0.6 g ascorbic acid ⁶	14 days ^{3,8}
Fluoride	P	Cool, 4°C	28 days ³
Hardness	P, G	HNO ₃ to pH < 2	6 months ³
Hydrogen ion (pH)	P, G	Cool, 4°C	Analyze immediately ³
Ignitibility	G	Cool, 4°C	28 days
Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
Nitrate	P, G	Cool, 4°C	48 hours ³
Nitrate-nitrite	P, G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
Nitrite	P, G	Cool, 4°C	48 hours ³
Odor	P, G	Cool, 4°C	24 hours ³
Oil and grease	G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
Organic carbon	P, G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours ³
Petroleum Hydrocarbons (TRPH)	G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
Phenols	G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
Phosphorus (elemental)	G	Cool, 4°C	48 hours ³
Phosphorus, total	P, G	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days ³
MBAS	P, G	Cool, 4°C	48 hours ³
Bromates (IC)	P, G	Cool, 4°C	30 days ³
Corrosivity (calculated)	P, G	Cool, 4°C	7 days ³
Residue, total	P, G	Cool, 4°C	7 days ³
Residue, filterable	P, G	Cool, 4°C	7 days ³
Residue, nonfilterable (TSS)	P, G	Cool, 4°C	7 days ³
Residue, settleable	P, G	Cool, 4°C	48 hours ³
Residue, volatile	P, G	Cool, 4°C	7 days ³
Silica	P	Cool, 4°C	28 days ³
Specific conductance	P, G	Cool, 4°C	28 days ³
Sulfate	P, G	Cool, 4°C	28 days ³
Sulfide	P, G	Cool, 4°C, add 2 mL zinc acetate plus NaOH to pH > 9	7 days ³
Sulfite	P, G	Cool, 4°C	Analyze immediately ³

Table 6-2. Required Containers, Preservation Techniques, and Holding Times
(Continued, Page 2 of 3)

Measurement	Container ¹	Preservation	Maximum Holding Time ² (Waters and Soils)
Temperature	P, G	Cool, 4°C	Analyze immediately ³
Turbidity	P, G	Cool, 4°C	48 hours ³
Organic Tests			
Carbamates	G, PTFE-faced silicone septum	Cool, 4°C Cl-CH ₂ COOH to pH <3	28 days ⁴
Glyphosate	G, Teflon®-lined cap	Cool, 4°C 0.008 % Na ₂ S ₂ O ₃ store in dark	14 days
Purgeable halocarbons	G, Teflon®-lined septum	Cool, 4°C, 0.008 % Na ₂ S ₂ O ₃ ^{5,6} store in dark	14 days
Purgeable aromatic hydrocarbons	G, Teflon®-lined septum	Cool, 4°C, 0.008 % Na ₂ S ₂ O ₃ ^{5,6} HCl to pH <2	14 days
Phenols	G, Teflon®-lined cap	Cool, 4°C, 0.008 % Na ₂ S ₂ O ₃ ⁵ store in dark	7/40 days for waters ⁴ 14/40 days for soils ⁴
Phthalate esters	G, Teflon®-lined cap	Cool, 4°C, store in dark	7/40 days for waters ⁴ 14/40 days for soils ⁴
PCBs, pesticides, herbicides	G, Teflon®-lined cap	Cool, 4°C, 0.008 % Na ₂ S ₂ O ₃ ⁵ store in dark	7/40 days for waters ⁴ 14/40 days for soils ⁴
Polynuclear aromatic hydrocarbons	G, Teflon®-lined cap	Cool, 4°C, 0.008 % Na ₂ S ₂ O ₃ ⁵ store in dark	7/40 days for waters ⁴ 14/40 days for soils ⁴
Volatile organics	G, Teflon®-lined septum	Cool, 4°C, 0.008 % Na ₂ SO ₃ ⁶ HCL to pH 2	14 days
EDB, DBCP	G, Teflon®-lined septum	Cool, 4°C, 0.008 % Na ₂ S ₂ O ₃ ⁶	28 days
Chlorinated hydro- carbons	G, Teflon®-lined cap	Cool, 4°C, store in dark	7/40 days for waters ⁴ 14/40 days for soils ⁴
Total organic halogens (TOX)	G, Teflon®-lined cap	Cool, 4°C, H ₂ SO ₄ to pH <2 store in dark	28 days ³
Acid and base/neutral extractables	G, Teflon®-lined cap	Cool, 4°C, 0.008 % Na ₂ S ₂ O ₃ ⁶ store in dark	7/40 days for waters ⁴ 14/40 days for soils ⁴
TCLP and ZHE extraction	P, G	Cool, 4°C	14/NS/14 days for VOCs 14/7/40 days for organics 180/NS/180 days for metals 28/NS/28 days for mercury
Wisconsin GRO	G, Teflon®-lined septum	Cool, 4°C, 500 uL 50% HCl (Water) Cool, 4°C, 25 mLs MeOH (Soil)	4 days shipping/14 days analysis 4 days shipping/14 days analysis
Wisconsin DRO	G, Teflon®-lined septum	Cool, 4°C, 5 mLs 50% HCl (Water) Cool, 4°C (Soil)	4 days shipping/47 days analysis ¹⁰ 4 days shipping/47 days analysis
Tissues			
Organics, inorganics tests	Aluminum foil and plastic bag	Freeze, -20°C or below	12 months
Metals tests	Plastic bag	Freeze, -20°C or below	12 months

Note: BOD = biochemical oxygen demand.
COD = chemical oxygen demand.
G = amber glass.
HCl = hydrochloric acid (metals grade).
HNO₃ = nitric acid (metals grade).
H₂SO₄ = sulfuric acid (metals grade).
NS = none specified by EPA.
MeOH = methanol.

Na₂SO₃ = sodium sulfite (ACS grade).
Na₂S₂O₃ = sodium thiosulfate (ACS grade).
P = polyethylene.
PCB = polychlorinated biphenyl.
NaOH = sodium hydroxide (ACS grade).
°C = degrees Celsius.
IC = ion chromatography.

Table 6-2. Required Containers, Preservation Techniques, and Holding Times
(Continued, Page 3 of 3)

- ¹ For nonvolatile organics, containers for soil and sediment samples are amber glass with Teflon®-lined caps and for volatiles, containers are amber glass with Teflon®-lined septum.
Soil sample containers for inorganics are amber glass jars with Teflon®-lined caps, polyethylene (P), or amber glass (G).
- ² Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the laboratory has data on file to show that the specific types of samples under study are stable for the longer time.
- ³ Holding times provided are for waters. EPA does not have holding times for these parameters in soil. These water holding times will be used as goals for those methods where a soil analysis is applicable.
- ⁴ 7/40 = 7 days until extraction; 40 days from extraction until analysis. 14/40 = 14 days until extraction; 40 days from extraction until analysis.
- ⁵ Sample preservation should be performed immediately upon sample collection. The only preservation for soil samples is cooling at 4°C. For composite samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed (maximum allowable time is 20 hours). $\text{Na}_2\text{S}_2\text{O}_3$ is used only in the presence of residual chlorine.
- ⁶ If residual chlorine is present, sodium thiosulfate is added to the sample vial. Note: It is not recommended to mix the two preservatives (and sample) together in an intermediate vessel.
- ⁷ These parameters are best analyzed in the field. In consideration of shipping limitations, when these analyses are requested of our laboratory for confirmation purposes, ESE's policy is to analyze these constituents within 24 hours of receipt.
- ⁸ The following test should be performed for cyanide samples:
 - (a) Oxidizing agents--Test the sample using KI-starch paper. If present, add a few crystals of ascorbic acid and test until negative. Add an additional 0.6 gram of ascorbic acid for each liter of sample to remove the chlorine.
 - (b) Sulfides--When sulfide is present as indicated by a positive test with lead acetate paper, the maximum holding time is 24 hours. Remove the sulfides by (1) filtration of sample if visible particulates are present, (2) precipitation with cadmium nitrate until a negative spot test is obtained, (3) filtration of the precipitate, and (4) addition of NaOH to pH > 12 if sulfides are not removed with the previous procedure.
- ⁹ Temperature and pH must be measured on-site at the time of sample collection. Seven days is the maximum time for laboratory analysis of total alkalinity, calcium ion, and total solids.
- ¹⁰ The holding time is the amount of time between receipt by the laboratory and addition of solvent to the sample. An exception will be allowed if samples arrive at the laboratory after 4:00 p.m. on a Friday. However, if the laboratory holds DRO samples over a weekend without adding the solvent to them, they must do so by 10:00 a.m. the following Monday. In no case may solvent be added past the 114 hours from the time of collection without flagging the data. It is not necessary for the laboratory to complete the extraction at the time of injection of the solvent.

Source: ESE.

6.5 REAGENT AND STANDARD STORAGE

Storage requirements of reagents and standards used are presented in Table 6-3.

Table 6-3. Reagent Storage

Reagent	Method of Storage*
Solvents	Stored in original containers in a vented storage room, or stored in double-walled flammable liquid storage cabinets. Stockroom personnel check the storage cabinets daily and transfer solvents from the storage room to the storage cabinets as needed. Note: Methanol used for volatile organic analyses are stored in the GC-Volatiles and GC/MS-Volatiles analysis areas. Acetonitrile, hexane, HPLC grade methanol, and MTBE are stored in the GC/HPLC analysis area.
Inorganic acids	Stored in original containers in the ESE stockroom. Once taken from the stockroom to a department, the acids are stored in the department's cabinet or under a fumehood.
Organic acids	Stored in original containers in the ESE stockroom. Once taken from the stockroom to a department, the acids are stored in the department's cabinet or under a fumehood.
Caustics	Stored in original containers in the ESE stockroom. Once taken from the stockroom to a department, the caustic reagents are stored in the department's cabinet or under a fumehood. Note: Caustic reagents are stored in separate cabinets from the acids.
Other reagents	Stored in the main chemical or standards storage room, or stored in the designated area in each department. Liquids in quantities of one gallon or more, not stored in a cabinet, must be kept in safety carriers. Standards that require storage at 4°C or at 0°C are stored in each department's refrigerators or freezers (respectively) designated for standards only.

Source: ESE.

* Once removed from the storage room or while in use, reagent bottles are kept in safety carriers.

7.0 SAMPLE CUSTODY

7.1 SAMPLE CUSTODY OBJECTIVES

The primary objective of sample custody is to create an accurate written verified record that can be used to trace the possession and handling of the samples from the moment of collection until receipt by the laboratory. Adequate sample custody in the laboratory are achieved by means of approved laboratory documentation.

7.1.1 DEFINITION OF LEGAL CHAIN OF CUSTODY

A sample for this project is defined to be in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked or otherwise sealed so that tampering will be evident; or
4. It is kept in a secure area, restricted to authorized personnel only.

7.1.2 LEGAL CUSTODY PROCEDURES

1. Formal chain of custody starts when the precleaned sample containers are dispatched to the field. The sample kit preparation personnel initiate custody of the sample containers by completing the first line under the "Relinquish By" of the Chain-of-Custody logsheet (Figure 7-3). Receipt of the sample containers is acknowledged by the field personnel by signing and dating the first line under the "Received By" on the Chain-of-Custody logsheet.
2. The formal Chain-of-Custody is signed by the Sample Custodian, or a designee, in the laboratory. In the field, the Field Team Leader or a designee is responsible to ensure that the Chain-of-Custody logsheet is maintained.
3. Copies of the Chain-of-Custody logsheets are maintained with project records.
4. Errors on all documents are corrected by striking one line through the error, then signing, and dating the corrections.
5. All documentation/logs are signed/initialed by appropriate personnel.

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Due to the evidentiary nature of the samples collected, possession of the Chain-of-Custody must be traceable from the time the sample containers leave the laboratory to the time they enter the field. Field chain of custody actually begins at the laboratory. Sample kits, which refer to coolers, sample containers, preservatives, and trip blanks are requested from the kit preparation staff using the Container Order Form (Figure 7-1). This form is completed by the Laboratory Coordinator or Project Manager and accompanied by the labels (Figure 7-4) and any other relevant information. Shipping labels are provided in accordance with current corporate policy on sample kit handling.

The pre-preserved sample containers (The bottles are labeled with the appropriate preservatives and preservation codes (Figure 7-5).); trip blanks, if needed; and Chain-of-Custody logsheet are packed in coolers, sealed, and shipped to the field personnel by bonded carrier (i.e., UPS or Federal Express). All Container Order Forms are signed and dated upon completion by kit preparation staff. The number of coolers shipped to the field is documented on the Container Order Form and on the shipping receipts. An ESE Cooler Tracking Report (Figure 7-2) indicating the personnel who prepared the kits, cooler number(s), project name and number, and contents of each cooler is generated. The Cooler Tracking Report is kept on file by Sample Receiving personnel.

7.1.3 DOCUMENTATION

The records for laboratory sample custody include:

1. Laboratory Forms:

- Container Order Form (Figure 7-1),
- Cooler Tracking Report (Figure 7-2),
- Chain-of-Custody Logsheets (Figure 7-3),
- Sample Label (Figure 7-4),
- Standardized Sample Preservation Codes (Figure 7-5),
- Sample Custody Logbook (Figure 7-6),

- Cold Room Sample Location Report (Figure 7-7),
Internal Chain-of-Custody (Figure 7-8),
Analysis Summary Form (Figure 7-9),
Internal Sample Arrival Notice (Figure 7-10),
VOA GC Sample Internal Chain-of-Custody (Figure 7-11), and
VOA GC/MS Sample Internal Chain-of-Custody (Figure 7-12).
2. Sample Extraction Log (Organic Laboratory/Extraction Logsheet, Figure 7-13,
Metals Laboratory/Digestion Logsheet, Figure 7-14).

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Figure 7-1 Container Order Form



Environmental
Science &
Engineering, Inc.

Labels _____

Container Order Form

Project Description: _____ Date: ____ / ____ / ____

Submitted By: _____ Ship To: _____

Project Manager: _____

Must Have Containers By: _____

Ship: ☐ Std. ☐ Overnight ☐ 2nd Day _____

# Samples	Matrix	Parameter(s)	Containers			
			Type	Size	#	Preservatives

Special Instructions:

	Yes	No
Chain of Custody	<input type="checkbox"/>	<input type="checkbox"/>
Blue Ice	<input type="checkbox"/>	<input type="checkbox"/>
Return Labels	<input type="checkbox"/>	<input type="checkbox"/>
Sampling Instructions (type)	<input type="checkbox"/>	<input type="checkbox"/>

Prepared By: _____

Date Sent: ____ / ____ / ____

Cooler #'s: _____

If there are any discrepancies, please contact ESE's Receiving Department at (800) 234-1239 immediately.

Figure 7-2 Cooler Tracking Report

Cooler #:

Prepared by:

On Hand: No

Ship to:

Client:

Proj Num:

Address

City

ZIP

Date Sent

Proj. Mgr.

Comments

ROTATION

Special Instructions:

Yes Chain of Custody (Y/N)

Yes Custody Labels (Y/N)

Yes Blue Ice (Y/N)

Yes Return Labels (Y/N)

No Sampling Instructions (Y/N)

Yes MSDS Enclosed (Y/N)

ST

Yes Std --

No Overnight -- SHIPPING

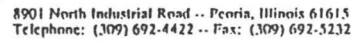
No Second Day --

No Three Day --

Sxs Matrix

Parameters

CONTAINERS			
Type	Size	#	Preservatives



FOR LAB USE ONLY

Project Number: _____

Due Date: _____

Nº 6125

[illegible]

SPECIAL INSTRUCTIONS:

Copies: White - Client Canary - Lab Receiving Pink - Lab File Goldenrod - Retained by Sampler

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Figure 7-3 Chain-of-Custody Logsheet

Figure 7-4 Sample Label

PRJ 391-5183 S402
FORT
IFFS01*1-C
SAMPLER DATE TIME
PH COND

PRJ 391-5183 S402
FORT
IFFS01*1-C
SAMPLER DATE TIME
PH COND

PRJ 391-5183 S402
FORT
IFFS01*1-EC
SAMPLER DATE TIME
PH COND

PRJ 391-5183 S402
FORT
IFFS01*1-Z
SAMPLER DATE TIME
PH COND

PRJ 391-5183 S402
FORT
IFFS01*1-S
SAMPLER DATE TIME
PH COND

PRJ 391-5183 S402
FORT
IFFS01*1-N
SAMPLER DATE TIME
PH COND

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Figure 7-7 Cold Room Sample Location Report

SEE COLD ROOM SAMPLE LOCATION REPORT FOR 05/24/94 04:32PM PAGE # 7

INITIALS FLD.GRP. FRAC LOCATIONS

INITIALS FLD.GRP. FRAC LOCATIONS

INITIALS FLD.GRP. FRAC LOCATIONS

17657	V	C-02	17658	SS	A-54	17659	SV	E-02
17659	EC	A-54	17659	V/C	D-02	17659	V/EC	A-50
17660	C	A-54	17660	O	A-54	17662	V/C	D-02
17662	V/EC	A-50	17663	N	A-54	17664	B	A-54
17664	C	A-54	17664	N	A-54	17665	SS	A-54
17666	C	A-54	17666	N	A-54	17667	N	A-54
17667	V	E-02	17668	N	A-54	17669	SS	A-54
17669	SV	E-02	17670	EC	A-54	17670	SS	A-54
17671	B	A-54	17671	N	A-54	17671	Z	A-54
17672	C	A-54	17673	B	A-01	17673	C	A-01
17673	N	A-01	17673	S	A-01	17673	X	A-01
17673	Z	A-01	17674	V	C-02	17675	SS	A-01
17675	SV	C-02 E-02	17676	O	A-01	17677	SV	C-01
17678	O	A-01	17679	SS	A-01	17680	B	A-01
17680	MS	A-01	17680	N	A-01	17680	O	A-01
17680	V	E-03	17680	Z	A-01	17681	SS	A-01
17682	C	A-01	17682	N	A-01	17682	N	A-01
17683	X	A-01	17684	EC	A-01	17684	V	C-03
17685	EC	A-01	17685	V	C-03	17686	SV	E-03
17686	V	E-03	17687	SS	A-01	17688	SS	A-01
17688	SV	C-03	17689	C	A-01	17689	C	A-01
17691	C	A-01	17691	N	A-01	17692	C	A-01
17692	O	A-01	17693	V	C-03	17694	C	A-01
17694	S	A-01	17695	SV	C-03	17696	AIR	A-01
17696	SS	A-01	17698	C	A-54	17698	MS	A-54
17698	N	A-54	17698	S	A-54	17698	Z	A-54

[illegible]

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Figure 7-9 Analysis Summary Form

Analysis Summary Form

Field Group: _____ Seq. #: _____ ESE Job #: _____

Date Received: _____ Turnaround Time: _____

Client Address: _____ Engineering-Related? Y/N _____

_____ Fax Results? Y/N _____

_____ If yes, Fax #: _____

ATTN: _____ Verbal Results? Y/N _____

DUE DATE: _____ If yes, Phone #: _____

1800	_____	18031	_____	1803KSCLP	_____	_____
1802C	_____	180310	_____	1803-AIR	_____	_____
1802C1	_____	180311	_____	1803-CHIPS	_____	_____
1802C1H	_____	180312	_____	1803-WIPES	_____	_____
1802C1S	_____	180313	_____	18041	_____	_____
1802C2H	_____	180314	_____	18042	_____	_____
1802C2S	_____	180315	_____	18043	_____	_____
1802C504	_____	18032	_____	18044	_____	_____
1802C505	_____	18033	_____	18045	_____	_____
1802C506	_____	18034	_____	1804D	_____	_____
1802C507	_____	18035	_____	1804D1	_____	_____
1802C508	_____	18036	_____	1804D2	_____	_____
1802C508A	_____	18037	_____	1804E	_____	_____
1802C5151	_____	18038	_____	1804I	_____	_____
1802CH	_____	18039	_____	1804M	_____	_____
1802CS	_____	1803H	_____	1804MI	_____	_____
1802CT	_____	1803HH	_____	1805A	_____	_____
1802F	_____	1803HHCLP	_____	1805AH	_____	_____
1802F1	_____	1803HS	_____	1805AS	_____	_____
1802F1H	_____	1803HSCLP	_____	1805AT	_____	_____
1802F1S	_____	1803J	_____	1805B	_____	_____
1802FF	_____	1803JA	_____	1805BH	_____	_____
1802FH	_____	1803JH	_____	1805BHCLP	_____	_____
1802FS	_____	1803JHCLP	_____	1805BS	_____	_____
1802G	_____	1803JS	_____	1805BSCLP	_____	_____
1802GS311	_____	1803JSCLP	_____	1805BT	_____	_____
1802GS50	_____	1803K	_____	180Z	_____	_____
1802GH	_____	1803KH	_____	351189	_____	_____
1802GHD	_____	1803KHCLP	_____	_____	_____	_____
1802GS	_____	1803KS	_____	_____	_____	_____

Total number of pages in this project: _____

Figure 7-10 Internal Sample Arrival Notice

Environmental Science and Engineering 01/18/95 STATUS:

P# 1 OF 1

*** SAMPLE ARRIVAL NOTICE FOR DEPARTMENT 18044 ***

C# 1 OF 1

PROJECT NUMBER
 FIELD GROUP
 COLLECTION DATES: A-01/18/95
 RECEIPT DATES :
 LAB DUE DATES : A-01/30/95
 TURNAROUND : 8
 QC :
 COMMENTS/QC FR: COOLER TEMP LIC ARM

PG NAME
 PROJECT MANAGER
 LAB COORDINATOR

MATRIX : WATER DET. LIMIT SPEC'D : NONE SPECIFIED
 SAMPLE FRACTIONS C, O, X

SAMPLE SEQ #
 COLLECTION DATE CODES: AAA
 RECEIPT DATE CODES: AAA
 LAB DUE DATE CODES: AAA

		EXTRACT	DET.LMT.
		GROUP	CRITERIA 123
001 - Oil & Grease	,MG/L	556-413.1-P	OCN

O-OPEN L-IN LAB E-EXTRACTED N-NOT REQUESTED D-DONE S-SCHEDULED
 - "STORET" - NOT ON DEPT AVAIL NUMS " " - SHORT HOLDING TIME

[illegible]

Figure 7-13 Organic Laboratory/Extraction Logsheet



Environmental
 Science &
 Engineering, Inc.

GC Sample Prep Log

Page _____

Date Extracted: ____/____/____

Extract Solvent: _____

Date Concentrated: ____/____/____

Extraction No.: _____

Extractors: _____

Cleanup: _____

Final Solvent: _____

Book No.: _____

	Client	Method No.	Sample ID	Initial/ Final pH	Sample Vol. (l)/ Weight (gm)	Surrogate Added		Spike Added		Final Vol. (ml)	Comments
						ID no.	Vol. (ml)	ID no.	Vol. (ml)		
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											

Comments _____

ATOMIC SPECTROSCOPY SAMPLE DIGESTION LOGBOOK

Batch No.: _____
Date: _____
Tech.: _____
Method: _____
Matrix: _____

[illegible]

Errors in all documents are corrected by following the procedure in Section 7.1.2.

7.2 FIELD CUSTODY PROCEDURE

To establish the documentation necessary to trace sample possession from the time of collection, a Chain-of-Custody record is completed and accompanies every sample. This record becomes especially important if the sample is to be introduced as evidence in court litigation. The record contains the following minimum information: sample description and matrix, analyses requested, signature of collector, date and time of collection, signature of persons involved in the chain of possession, and comments such as suspected hazards or visible/suspected physical characteristics of the sample.

In collecting samples for evidence, only the number of samples which provides a good representation of the media being sampled are taken. To the extent possible, the quantity and types of samples and sample locations are determined prior to the actual field work. As few people as possible handle the samples. The samples are under the direct control of the field sampler for that project.

The field samplers are personally responsible for the care and custody of the samples collected until they are transferred or dispatched properly.

Sample labels are completed for each sample using waterproof ink, unless prohibited by weather or other special conditions. For example, a logbook notation could explain that a pencil was used to fill out the sample label because a ballpoint pen would not function in freezing weather. Labels are affixed to sample containers prior to the time of sampling. The labels are filled out at the time of sampling.

The field supervisor determines whether proper custody procedures were followed during the

field work and decides if additional samples are required.

If at any time the samples are to leave the immediate and direct control of the field sampler prior to delivery to ESE, cooler seals are used to detect unauthorized tampering. Cooler seals are gummed paper or similar material. The paper seal includes the following minimum information: Collector's name, date, and time of sampling, identifying number or reference.

The cooler seal should be attached in such a way that is necessary to break it in order to open the shipping container. Seals are affixed to the containers before the samples leave the custody of sampling personnel unless the samples are transferred directly from the field sampler to the authorized Sample Custodian of ESE.

7.3 TRANSFER OF CUSTODY AND SHIPMENT - FIELD TO LABORATORY

Samples are delivered to ESE for analysis as soon as practical - usually within one or two days after sampling. The samples are accompanied by the Chain-of-Custody completed by the field sampler at the time of collection and delivered to the Sample Custodian or the designee.

When transferring the possession of samples, the individuals relinquishing and receiving shall sign, date, and note the time on the Chain-of-Custody. This record documents sample custody transfer from the sampler, to the laboratory and subsequently, sample storage. Each individual who signs the Chain-of-Custody has a responsibility to ensure that all information added to the Chain-of-Custody is complete and accurate.

Samples are packaged properly for shipment (including custody seals) and dispatched to ESE for analysis, with a separate Chain-of-Custody accompanying each shipment (each ice chest). The method of shipment, courier name(s), and other pertinent information is entered into the "Comments" section of the Chain-of-Custody.

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7.4 LABORATORY CUSTODY

Sample chests (packages/coolers) are transported to the laboratory. The Sample Custodian, or designee, then signs the Chain-of-Custody indicating receipt of the samples by the laboratory. The Sample Custodian records the samples as having been received by the laboratory in the Sample Custody Logbook (Figure 7-6). The information recorded includes sample receipt date, morning or afternoon designation, sample identification (client), the number of samples received, unique laboratory identification number, the analysis due date, the sample carrier (e.g. UPS, Federal Express), the sampling date, analyses requested, and the sample matrix (matrices).

The samples are checked in by the Sample Custodian for proper preservation (e.g. pH, temperature), integrity (e.g., leaking, broken bottles, tainted custody seals), and proper, complete sample documentation and identification. Sample chests or coolers that are not within the 4 ± 2 degrees Celsius ($^{\circ}\text{C}$) requirement are reported immediately to the Project Manager to determine if resampling will be required. All samples contained in the shipment are compared to the Chain-of-Custody to ensure that all samples designated on the custody record have been received. The Sample Custodian notes on the Chain-of-Custody any special remarks concerning the shipment. Any marks or notes made on the Chain-of-Custody document by the Sample Custodian are clearly distinguished from original field notations. The Sample Custodian reviews the integrity of all sample fraction containers and checks the accuracy and clarity of all documentation received. The Sample Custodian audits daily the first shipment of representative samples of all fractions requiring field preservation to ensure that they have been properly preserved. The audit is recorded in the Receiving pH logbook. The Sample Custodian preserves unpreserved fractions or adds additional preservative, if needed, upon receipt. Deficiencies in sample preservation, additional preservative added, and all other inadequacies are recorded on the Chain-of-Custody and reported to the Project Manager. The Project Manager, upon consultation with the client/field team, decides if resampling is required. The original Chain-of-Custody is sent back to the client with the final report. A copy of the Chain-of-Custody is kept in the internal project file with a copy of the final report.

The accepted samples are logged into the ESE laboratory LIMS (Laboratory Information Management System), CLASS™ (Section 7.5) using the unique laboratory sample identifications, which includes the ESE project identification number and sample ID provided by the sampler on the Chain-of-Custody. The sample collection date and receipt date are recorded and are used for monitoring holding time and progress of the project throughout the laboratory. The requested analyses are assigned to the individual samples and sample arrival notices (Figure 7-10), which are used for internal project tracking, are generated. The arrival notices are distributed to the appropriate laboratory sections by the Project Coordinator to notify the analysts of the arrival of the samples, identification and number of samples, required analyses, due dates, and specific QC requirements. Any special instructions or notes listed on the Chain-of-Custody will be mentioned on the arrival notice. To facilitate intralaboratory communication, the Sample Custodian who logged in the samples and the Project Manager for the project are recorded on the arrival notice. The arrival notices with the attached preparation logbook pages for samples requiring preparation before analyses, such as organic extractions and metals digestions, are forwarded to the appropriate analytical instrument section after the preparation has been completed. Any problems or observations noted during the preparation process are recorded on the arrival notice and entered into CLASS™. An Analysis Summary Form (Figure 7-9) is also created and filed in the project folder to track which departments received samples. Upon completion of the analyses required for the samples, the arrival notices are returned to the project folder and their completion noted on the Analysis Summary Form. The final report is then generated by an Administrative Assistant. Tracking the samples through the laboratory is done by the Work-in-Progress report which is distributed to project management, operations management, department management, and QA/QC. The Work-in-Progress report, created by an Administrative Assistant, is a daily register of all samples within the laboratory, listing the client name, project identification number, number of samples for that project, date received, departments receiving the samples, due date, and status (for example, a rush status could be listed).

Samples are placed in appropriate storage areas in the laboratory depending on storage requirements. The majority of the samples are stored in the main coldroom, with the exception

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of volatile samples. The samples in this storage area are arranged by field group. The main coldroom is refrigerated at $4 \pm 2^{\circ}\text{C}$ and kept locked after normal working hours. All volatile samples are refrigerated at $4 \pm 2^{\circ}\text{C}$ and stored in the GC and GC/MS Volatiles area. An Internal Chain-of-Custody is maintained for the GC volatiles refrigeration unit (Figure 7-11) and GC/MS volatiles refrigeration unit (Figure 7-12) for all projects, since volatile samples are not stored in the main coldroom and are transferred to the appropriate volatile units. If requested by a client, an Internal Chain-of-Custody is maintained for the main coldroom (Figure 7-8). Then, when an analysis is scheduled, the analyst will request the samples of interest from the Sample Custodian. The Sample Custodian will, in turn, remove the samples from storage and sign over the custody of the samples to the analyst. Both individuals will place their signatures, along with the date and time of transfer on the internal chain of custody form maintained by the Sample Custodian. At that moment, the analyst accepts custody of the samples. When the analysis is complete, the analyst will return the samples and their custody to the Sample Custodian with signature, date and time on the custody form. Sample digestates and extracts also require a chain-of-custody. Custody of the extract or digestate begins at the moment of preparation and is documented on the sample extract or sample digestion logs. A particular analyst or the laboratory section manager maintains custody of the sample extracts or digestates until they are transferred to another analyst or section for analysis, storage, or disposal. A Cold Room Sample Location Report (Figure 7-7) is generated weekly to facilitate sample retrieval. Sample storage areas are used only for sample storage. Samples remain in storage for one month after receipt into the laboratory unless otherwise directed by the client. Sample extracts remain in storage for one month after analyses unless otherwise directed by the client.

During normal work hours, there are always laboratory staff present in the ESE Peoria Laboratory. Entry to the building for visitors is available through the front door of the main building or the ESE Peoria Laboratory receiving area located at the north side of the main building. A receptionist is present at the front door to greet visitors. Visitors must sign a visitor's register and are escorted through the building by ESE personnel. The building is continuously locked and is secured with a Security Link® Alarm System after normal working

hours.

When it is necessary to use another laboratory for sample analysis, the Project Manager is responsible for arrangements with the second laboratory. The samples are only subcontracted to a state or federal government agency, or client-approved laboratory. The Chain-of-Custody accompanies samples transferred to another laboratory and includes the following information: collection data and time, field ID, laboratory ID, date of sample preparation, and requested analyses.

The samples are kept at $4 \pm 2^{\circ}\text{C}$ prior to and during shipment. A Chain-of-Custody indicating samples and fractions sent accompany the samples to the subcontractor. The subcontractor signs and dates the Chain-of-Custody upon receipt of the samples. A copy of the signed Chain-of-Custody is returned to ESE and placed in the project file.

7.5 LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS)

CLASS™ is an automated, in-house-developed LIMS that integrates information from sample collection, laboratory analyses, and QC requirements; and calculates, checks, stores, and reports data in a variety of formats. CLASS™ resides on a fileserver using Novell Netware version 3.12, and contains 1.6 gigabytes of storage. In Peoria, the network is connected to more than forty personal computers, and via the Wide Area Network, connected to all other ESE laboratories and engineering facilities. CLASS™ is managed by the Laboratory Information Services Department within the Peoria Laboratory, with support from the ESE Gainesville Laboratory Information Services Department. All data from analyses performed by the laboratory are managed and stored using CLASS™.

The database is stored, processed, and retrieved using the database manager Advanced Revelation® (copyright Revelation Technologies). The file structure and indexing provided by Advanced Revelation® allow easy retrieval, grouping, and formatting of data. Incorporated into

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the system is the ability to combine field data, analytical results, and QC data and produce specially formatted project-specific reports, statistical analyses, plots, and electronic files.

CLASS™ manages the flow of samples and data through the laboratory. The Project Manager provides information on the number of samples, site IDs, parameters to be analyzed, and estimated collection dates prior to sampling, if applicable. This information is entered into CLASS™ and used to produce sample labels. A unique ESE number is assigned to each sample, and labels with that number and the site ID are placed on each container for that sample. At each site, samples are collected and placed in the appropriate pre-labeled containers. Sampling information is recorded on the Chain-of-Custody. Samples accompanied by the Chain-of-Custody are sent to the laboratory where they are checked, processed, and stored by the Sample Custodian. The samples, along with the date of collection and site identification, are logged into CLASS™ by the Sample Custodian. Chain-of-Custody forms are placed in the project file and maintained by the Laboratory Coordinator.

ESE uses a combination of EPA Storage and Retrieval (STORET) numbers and company-assigned Method Codes to designate parameters required for analysis. Each STORET-method combination has its own laboratory QC requirements specific to that analytical method stored in CLASS™. A list of all required parameters is logged into the computer with each sample. This list is identified on the sample arrival notice for each sample.

The sampling information is entered into the computer to activate the parameter list for the samples collected and received by the laboratory. A report (Available Numbers) of samples available for each analysis indicates the number of days left before the holding time is exceeded for each method for each sample. This report is regularly produced and distributed to each laboratory department.

CLASS™ uses a batch method for analyzing, checking QC, and calculating final results of samples. Prior to analyzing a sample batch, the analyst designates a specified group of samples

in the computer and the sample-parameter status is updated. The analytical batch is assigned a unique batch control number, which is stored with all final data, to facilitate data review, QC reporting, and retrieval of original documentation.

The production of each laboratory batch usually requires several distinct activities. Usually, instrument calibrations are entered first and include several QC checks by CLASS™. The linear (or quadratic) regression equation and correlation coefficient are calculated from the calibration curve data, and the correlation coefficient is tested to determine whether it is within an acceptable range specific to the analysis. Method blank and control spike information are then entered, and results are calculated and checked against control limits for that method. Sample responses are entered into the batch, and final concentrations are calculated for each sample. Responses are checked to ensure that they are bracketed by the standard curve. The batch printout includes a QC summary showing the automated QC checks, such as holding times, the presence of spikes, and acceptable spike recoveries. Any discrepancies are flagged by the computer for the analyst.

The batch printout also documents that the analyst has checked data entries and provided all required documentation for the analysis. The batch printout is completed, signed, and dated by the analyst. The batch along with the raw data are reviewed and signed by the Department Manager or a designated reviewer.

The Department Manager or designated reviewer processes the batch in the computer to verify QC and to update the sample records and final calculated concentrations. Once a batch has been finalized by the Department Manager or reviewer, the batch is locked and data cannot be changed. The final report is then generated and reviewed by the ESE Project Manager before it is sent to the client. If batch edits are required, the LIMS Manager is notified and definalizes the batch. Changes and refinalization are done by the appropriate Department Manager. The original and revised batch reports are found in the batch folder, along with documentation concerning the reason for the batch definalization.

Each employee is assigned an individual access code for entry into CLASS™. All personnel with an access code may retrieve information from the system. Access rights are assigned on an individual basis. Laboratory personnel are not allowed to update sample records without authorization from the LIMS Manager. Only personnel with appropriate access codes and LIMS Manager approval may edit laboratory data.

The batch folders, with all supporting documentation (such as organic extraction log pages (Figure 7-13) and metals digestion log pages (Figure 7-14)), are filed chronologically by department in a secured Information Services storage room; file cabinets with project files are stored similarly. These may be signed out for review by the analysts, Project Coordinators, Project Managers, Department Managers, or QA/QC personnel. A Document Control Logbook (Figure 12-3) is used to track folders that have been checked out. Batch folders and project files are kept a minimum of ten years.

Laboratory personnel use the computer to monitor the flow of data through the system. Data are accessed and reported by sampling event, project, or any subset of samples and parameters.

CLASS™ enables a Laboratory Coordinator or Administrative Assistant to:

1. Produce a variety of summary reports of analytical data,
2. Produce sample summary reports,
3. Calculate statistics such as mean, maximum, minimum, and standard deviation,
4. Summarize QC in various formats, and
5. Produce a project-specific export-data file.

Data are stored in the CLASS™ database and can be exported electronically into Lotus and DBASE files. Many client-requested formats have been developed in CLASS™ for electronic data transfer. When a client requests an electronic data transfer, a regular hardcopy data report is usually sent in addition to the electronic file. Copies of both electronic and hard copies are maintained in project files.

Information Services supports a staff of computer programmers to maintain and modify CLASS™. Requests for new programs or changes are kept in both electronic and hardcopy files; the name of the person making the request and the programmer are included. Every change made to a program is documented electronically at the end of the program with the date, employee number of the programmer, and a brief description of the change. A summary of these changes is maintained in CLASS™ listing the programs, changes, requestors, and programmers. All program revisions are documented in a revisions file and can be reviewed anytime. Completed requests are tested by the programmer staff and then verified by the requestor.

The QA staff checks data packages quarterly, including computer printouts, to verify that CLASS™ data match raw data from the laboratory.

The database is backed up daily except Saturday using high-density storage media. The tapes are stored in the Information Services air-conditioned locked office.

8.0 ANALYTICAL PROCEDURES

8.1 STANDARD PROCEDURES

Standard analytical procedures to be used for any project for chemical analysis of water and soil are referenced in Section 5.0. Laboratory Department Managers will ensure that only these standard analytical methods are employed by the staff. Standard operating procedures are required for all departments and development of the documents are ultimately the responsibility of the Department Managers. The methods cited in these documents are the methods normally used. Any deviation from the standard method is documented in the analyst notebook and approved by the Department Manager.

For parameters not listed, nonstandard methods may be specified by the client or developed by the laboratory. Nonstandard methods are validated as described in Section 8.2.

8.2 NONSTANDARD METHODS VALIDATION

If other than standard analytical methods become necessary due to a change in work scope, it is necessary to validate the analytical method. Method validation is warranted when major modifications of standard methods such as extraction, preparation, and cleanup procedures and/or the application of a standard method to new analytes or matrices. The responsible Department Manager or analyst must establish a thorough method validation so that the selected method measures the reported parameter with the necessary precision, accuracy, and detection limit, without severe interference by other constituents in the sample. If required, nonstandard methods and validation documentation will be submitted to state or government agencies (i.e. IEPA, USACE, etc.) and clients for review and approval prior to use on samples for analyses.

The requirements for method validation include the performance of an Initial Demonstration of Capability and Method Detection Limit Study. The following

subsections constitute the minimum requirements for initial establishment of the accuracy, precision, and detection limits of nonstandard methods.

8.2.1 INITIAL DEMONSTRATION OF CAPABILITY

For each parameter of interest, a minimum of four replicate spike samples are prepared from laboratory blank water at one appropriate analyte concentration. Spiked samples are analyzed according to the method. An unspiked "standard" matrix blank or unspiked laboratory blank water is analyzed. The spiking concentration is selected such that the final extract or aliquot is analyzed in the midrange of the calibration curve.

The Initial Demonstration of Capability protocol is summarized below:

Accuracy (Recovery) The minimum requirements for establishment of accuracy for methods are as follows:

1. Calculate the found concentration for each spiked sample as follows:

R = measured concentration = measured concentration in spiked sample
minus the measured concentration in unspiked (blank) sample.

2. Calculate the Percent Recovery (P) for each spiked sample as follows:

$$P = \frac{R}{S} \times 100$$

where: R = measured concentration for each spiked sample
 S = target concentration for each spiked sample.

3. Calculate the Average Percent Recovery (P_{av}), Standard Deviation of the percent recoveries (S_r), and Percent Relative Standard Deviation of the percent recoveries (RS_r) of the spiked samples as follows:

$$P_{ave} = \frac{P_1 + P_2 + P_3}{3}$$

where: P_1 , P_2 , and P_3 = percent recovery of the three spiked samples

$$S_r = \sqrt{\frac{1}{n-1} \left[\sum_{i=1}^n R_i^2 - \frac{1}{n} \left(\sum_{i=1}^n R_i \right)^2 \right]}$$

where: S_r = standard deviation of P_{ave}

$$RS_r = \frac{S_r}{P} \times 100$$

where: n = number of recovery values, and
 RS_r = relative standard deviation of P .

Precision The minimum requirements for establishment of precision for methods are as follows:

1. Calculate the Relative Percent Difference (RPD) between each pair of replicate spiked samples.

$$RPD_1 = \frac{|R_1 - R_2|}{(R_1 + R_2)/2} \times 100$$

$$RPD_2 = \frac{|R_1 - R_3|}{(R_1 + R_3)/2} \times 100$$

$$RPD_3 = \frac{|R_2 - R_3|}{(R_2 + R_3)/2} \times 100$$

2. Calculate the average RPD for the spiked samples.

$$RPD = \frac{RPD_1 + RPD_2 + RPD_3}{3}$$

8.2.2 METHOD DETECTION LIMIT

The detection limit of the method is the lowest sample concentration that can be reliably recovered and measured in the sample matrix with a low background level. Statistically based procedures to determine absolute method detection limits (MDLs) as described in 40 CFR Part 136 Appendix B are used. For each parameter of interest, a minimum of seven replicate spike samples are prepared from laboratory blank water at one appropriate analyte concentration. Spiked samples are analyzed according to the method. An unspiked "standard" matrix blank or unspiked laboratory blank water are analyzed. The spiking concentration is selected such that the concentration is approximately one to ten times the estimated or method detection limit for the parameter.

The reported detection limit for a method is subject to the judgment of the analyst and the Department Manager and takes into account background levels, instrument baseline noise, spiking recoveries, and the lowest calibration standards analyzed. In general, (except for those methods where the detection limit is derived from instrument considerations), the reported detection limit for a method is determined by the lowest standard concentration analyzed, taking into consideration the sample volume or weight of sample used and the final extract volume (where applicable).

Method validation determination results (Initial Demonstration of Capability and Method Detection Limit studies) are recorded and submitted to the Department Manager and Laboratory QA/QC Coordinator prior to the initiation of analysis. Before analysis begins, the Department Manager assures that the method meets the performance criteria required by the project.

Once the method is validated, the initial validation data (precision and accuracy) are periodically revised, updated, and improved using the data acquired during the laboratory's routine analytical QC program.

8.3 LABORATORY GLASSWARE

Dirty glassware is drained of solvents and rinsed with tap water when soils or other residues are still remaining, before it is washed.

All laboratory glassware (i.e., volumetric flasks, separatory funnels, beakers, graduated cylinders, etc.) is cleaned according to the analysis/parameter group listed in Table 8-1. These cleaning procedures are subject to change depending on the requirements of the projects.

8.4 LABORATORY METHOD MODIFICATIONS

Laboratory method modifications are done either to improve the method efficiency or add new compounds to an approved method. ESE has several method modifications involving the addition of new compounds to a specific EPA method(s). These compounds are denoted and their QA targets found in Section 5.0. Initial Demonstrations of Capability and Method Detection Limit studies were performed for the compounds.

8.5 REAGENT STORAGE

The procedures for storing reagents in the laboratory are presented in Section 6.5. All reagents are marked with initials, date received, and date opened.

Table 8-1. Glassware Cleaning Procedures

Analysis/Parameter	Cleaning Protocol*
Extractable Organics	1,2,3,4,5,8,9
Purgeable Organics (Volatiles)	5,4,8
Trace Metals	1,2,3,5,6
Nutrients, Minerals, Demands, Cyanide, Phenols	1,2,3,5
Gravimetric, e.g. Residues, Oil and Grease	1,2,3,5,8
Phosphorus, All forms	1,2,3,7,5

Note: HCl = Hydrochloric acid
HNO₃ = Nitric acid

*Cleaning Procedures

1. Remove all labels using sponge or brush.
2. Wash with hot soapy water (use Liquinox soap only) using brushes to scrub inside of glassware, stopcocks, and other small pieces if possible.
3. Rinse three times with tap water.
4. Rinse three times with histological grade methanol.
5. Rinse three times with deionized water.
6. Acid rinse with dilute HNO₃ and then with tap water.
7. Acid rinse with 1:1 HCl and then with tap water.
8. Bake at 180°C for 1 hour or until dry.*
9. Rinse with appropriate extraction solvent prior to use.

* Class A volumetric glassware should not be baked.

Source: ESE.

8.6 LABORATORY WASTE DISPOSAL

It is important that all waste materials generated in the laboratory be disposed promptly and properly. The following subsections describe the procedures for handling laboratory waste.

8.6.1 LIQUID WASTES

In general, no chemical wastes are disposed in the sinks without contacting the Department Manager or Hazardous Waste Coordinator (HWC). Only certain dilute acid wastes are disposed in the sinks.

8.6.1.1 Acid Wastes

All acid waste (not containing heavy metal concentrations to be considered a "regulated waste") generated by the Atomic Spectroscopy and Water Quality Department as digestates and instrument waste are disposed in the designated Acid Waste plastic drum located in the Metals Digestion area and the digestion tubes discarded. All TCLP extracts are disposed in the designated Acid Waste containers located in the Metals Digestion and Water Quality areas.

8.6.1.2 Disposal of Standards and Solutions

As standards and solutions are made, the solvent, constituents, date prepared, expiration date, reference number, and initials of preparer must be put on the container. This information must be on the container before it is disposed by the HWC. Standards containing any amount of organic solvent are not poured down the sink. Aqueous standards containing organic or inorganic (metals, etc.) compounds are either disposed in the appropriate waste drum, or picked up by the HWC.

8.6.1.3 Disposal of Solvent Wastes

All waste solvents are disposed in approved solvent waste containers located throughout the departments in the laboratory. Solvents are segregated according to the designated chemical types and placed only in the appropriate waste container. The waste containers are emptied

on a regular basis by the HWC. If the containers become full before then, the HWC will be called so the containers can be emptied.

Solvents will be segregated as follows:

Pentane/Fuel: Fuel, oil, pentane

Freon: 1,1,2-Trichlorotrifluoroethane only

Chlorinated: Methylene chloride, chloroform

Non-Chlorinated

Flammable: Acetone, benzene, cyclohexane, ethyl ether, hexane, isopropanol, petroleum ether, toluene, xylenes, MTBE, carbon disulfide

HPLC: Acetonitrile, isopropanol, methanol, ethanol, water, carbamate analysis waste (OPA, NaOH, sodium borate, 2-mercaptoethanol)

Mixed Chlorinated

Flammable: Mix of methylene chloride, acetone, hexane, ethyl ether

Isopropanol may be disposed in either the Non-Chlorinated Flammable or HPLC container. Freon-112 is disposed only in designated waste containers in the Water Quality department, and not combined with other chlorinated wastes. Glass solvent containers are not accepted for solvent waste. Solvents are segregated as described and placed in designated waste containers.

8.6.1.4 Disposal of Extracted Water Samples

Water samples which have been solvent extracted are neutralized and disposed in the designated Sample Preparation department waste sink.

8.6.1.5 Disposal of Inorganic Wastewater

Samples and waste generated from the analysis of inorganic parameters such as phenols and cyanide are disposed in the designated waste containers in the Water Quality department.

8.6.2 SOLID WASTES

8.6.2.1 Solvent Saturated Soil and Solids

Non-hazardous waste such as packing materials, tape, or plastic wrap are disposed in trash receptacles. Cardboard, paper, aluminum, glass (not for laboratory use), or polyethylene plastic is recycled and is taken to the Sample Receiving department. Broken laboratory glass is placed in the repair box or disposed in the designated broken glass containers. Solvent saturated solids and filters, such as sodium sulfate or soil saturated with methylene chloride, are never disposed in trash receptacles. These solid wastes are placed in solid waste cans located in each department. These waste cans have lids to prevent fumes from entering the laboratory air. Full containers are collected by the HWC on a regular basis. The soil waste is taken to the HazWaste Area and placed in the Soil Trough. The contents of the Soil Trough will be allowed to air dry and disposed in the dumpster by the HWC.

8.6.2.2 Disposal of Expired or Contaminated Chemicals

Commercial chemicals, solvents, and standards that are out of date or contaminated are left in their original containers. The container is labeled, including the date and initials, prior to disposal. The HWC is then called to pick up the material.

8.6.2.3 Disposal of Autosample Vials Containing Extracts

All autosample vials are collected in the designated containers in each department. The containers are emptied on a regular basis by the HWC. If the container becomes full, the HWC is called.

8.6.2.4 Disposal of Additional Hazardous Material

The contents are clearly marked on the container or on an accompanying analysis report. The containers are dated and initialed. The HWC is contacted for pickup and disposal.

8.6.3 UNKNOWN WASTES

If an unmarked container or unknown waste is found, it is brought to the attention of the Department Manager or HWC. Unknowns are not allowed to accumulate. Unknowns are identified prior to disposal.

8.6.4 SAMPLE WASTES

After the completion of a project, an Administrative Assistant generates two final reports. The Project Manager sends the original to the client and indicates on the duplicate report whether to "Dispose", "Hold" and "Dispose On", or classify as "Hazardous" the samples for that project. The report is then distributed to the Sample Receiving Technician (SRT) who coordinates sample disposal. If the samples cannot be disposed due to recommendations or requirements and the date is after the six week sample shelf-life, the Project Manager marks "Hold" and a "Dispose On" date on the report to indicate which samples are to be retained. If there is no indication the samples are a regulated waste or pose a hazard to individuals working with the samples, the Project Manager marks "Dispose" on the report, indicating which samples are to be disposed. Ultimately, the Project Manager signs and dates the report authorizing disposal. If the samples are not regulated as a waste, but the samples pose a hazard to individuals handling them (odorous, etc.), the Project Manager indicates the concern on the report. The Project Manager then signs and dates the report to authorize disposal. If the samples are considered "Hazardous Waste", the Project Manager indicates and highlights "Hazardous" on the report. The Project Manager signs and dates the report authorizing disposal. Once the report is received by the SRT, the appropriate samples are removed the storage unit. If the samples are not "Hazardous" and not a regulated waste per the specified analysis, the SRT bulks the samples together for disposal.

8.6.4.1 Disposal of Water Samples

All water samples are neutralized and disposed by the SRT. The empty containers are recycled. All labels are removed prior to disposal. Appropriate protective equipment is worn when handling samples and containers.

8.6.4.2 Disposal of Soil/Solid Samples

All soil/solid samples are disposed by emptying the contents in the Soil Waste Drum in Sample Receiving. All labels are removed prior to disposal. Appropriate protective equipment is worn when handling samples and containers.

8.6.4.3 Disposal of Hazardous Samples

If the samples are regulated or classified as "Hazardous", the samples are either disposed or lab-packed by the HWC, depending on the classification. The HWC stores the samples in the designated sample waste area. The analysis report is kept in the HWC's waste files until the next hazardous waste pickup. During the storage time, the HWC combines all compatible samples to achieve the smallest overall volume.

9.0 CALIBRATION PROCEDURES AND FREQUENCY

Calibration procedures establish the relationship between a calibration standard(s) and the measurement of that standard by an instrument or analytical procedure. At a minimum, calibration is required: (1) when an analytical method is first set up, (2) when the instrument detector has been subject to major maintenance, or (3) when the instrument fails the calibration QC checks.

All analytical instruments are calibrated with a series of standards. The series of standard solutions is prepared from stock standards. These standards are either purchased from various vendors in premixed solutions or prepared directly from stock compounds. The preparation of all standard solutions is documented in logbooks. All stock standards are inscribed with date received, date opened, date prepared (laboratory), and expiration date. The standards are stored in designated areas and checked for expiration on a regular schedule. Specific calibration requirements for major classes of analytical procedures are described in the following sections.

9.1 STANDARD RECEIPT AND TRACEABILITY

A standard is a solution of an analyte of interest with verifiable accuracy which is used to evaluate that constituent in a sample. Before any standard is purchased from a supplier, traceability and safety must be considered. This includes a consideration of the standards purity. The purity of the target compound must be verified and the accuracy requirements for its measurement available. The manufacturer ensures this through certification and traceability statements, which are kept on file in the laboratory. All laboratory standards must be traceable to a NIST (or EPA equivalent) source. Other chemicals must have a purity specification mentioned on their labels. The safety requirements are checked with the material safety data sheets (MSDS), supplied by the manufacturer.

Upon receipt, the standard is cross referenced with its purchase order to confirm that what was received is what was ordered. The chemical is checked with the purchase order and is placed on a table in a central area that is checked daily by the Department Manager. The standard receipt date is noted on each standard. All standards are stored in designated areas for each department.

9.2 STANDARD SOURCES AND PREPARATION

All standards must be traceable to a reference source to meet the accuracy requirements as outlined in Section 5.0. The concentrations of the working solutions will depend on the calibration range of each analyte of interest. All initial standard preparations are recorded in appropriate logbooks. The information recorded is the standard prepared, the source and concentration of the standard, the expiration date of the neat chemical used to prepare the standard, the standard lot number, date prepared, and initials of the preparer. The protocol for standard sources and preparation is located in Table 9-1. All standards should not exceed the storage (use) life of both the stock and working solutions. Each working solution and stock solution is labeled with date prepared, expiration date, initials, concentration used, and reference number of the appropriate standard preparation logbook.

Secondary dilutions made from stock standards are also recorded in a logbook. The lot number of the stock standard used and the notebook number and/or page number are also indicated in the logbook for traceability. Table 9-1 lists the frequency of standard preparation and storage of standards by instrument group.

9.3 LABORATORY INSTRUMENTS

All laboratory instrumentation is listed in Table 9-2. Since calibration criteria is required for analytical operations, each of these instruments are calibrated in a manner consistent with EPA calibration protocols and/or ESE SOPs. Calibration is documented in an analysis logbook.

Table 9-1 Standard Sources and Preparation

Instrument Group	Standard Source(s)	Condition Received	Storage	Standard Preparation	Lab Stock Storage	Preparation Frequency
ICAP GFAA CVAA FLAA	Various	1,000 or 10,000 ppm soln.	RT	Intermediate and/or Working Stock	RT	> 1 ppm Monthly < 1 ppm Daily (GFAA)
Autoanalyzer	Various	Neat and/or Solution	RT	Primary Intermediate Working	RT RT RT	Monthly Biweekly Biweekly
IR	Various	Neat (oil) and/or Prepared	RT	Combined Primary Intermediate Working	RT RT RT	Quarterly Monthly Monthly
Inorganics	Various	Neat and/or Prepared	RT	Primary Working	RT RT	Quarterly Daily
GC (non-VOA)	Various	Neat, Mix, and/or Prepared	Freezer and/or Refrig.	Primary Intermediate Working	Freez/Ref Refrig. Refrig.	Semiannual Method Specific Monthly
GC (VOA)	Various (Ultra- Scientific)	Neat Mix Solution Solution	Freezer Freezer Freezer	Mixed Primary Intermediate Working Working Mixed Primary Intermediate Working	Freezer Freezer Freezer Freezer Freezer Freezer	Monthly Weekly Daily Weekly Monthly Weekly Daily
LC	Various	Neat Mix Solution	Freez/Ref Freez/Ref	Primary Intermediate Working Primary Intermediate Working	Refrig. Refrig. Refrig. Refrig. Refrig.	Semiannual Monthly Biweekly Semiannual Monthly Biweekly
GC/MS (non-VOA)	Various	Mix Solution	Freez/Ref	Working	Freez/Ref	Semiannual
GC/MS (VOA)	Various (Supelco)	Mix Solution	Freezer	Working	Freezer	Biweekly

Note: RT = Room Temperature
IR = Infrared
Freez = Freezer
Ref = Refrigerator

Source: ESE.

Table 9-2. List of Laboratory Instruments

Analysis Type	Number	Instrument
Gas Chromatography/ Mass Spectrometry: Semivolatiles	1	HP 5971 GC/MS with a HP 5890 GC capillary direct with HP-7673 Autosampler; instrument uses a HP/UX computer with a Target Operating system for data acquisition and reduction.
	1	EXTREL ELQ-400 MS with a Varian 3400 capillary direct GC with Leap A-200S Autosampler; utilizes NASTech software for data acquisition and reduction.
Volatiles	3	EXTREL ELQ-400 MS with Varian 3400 capillary direct GCs with jet separator interface; each instrument is attached to a Tekmar 2000 liquid sampler (LCS) and Tekmar 2016 or 2032 sixteen position autosamplers (ALS); all three instruments have DEC RSX-11M computers with NASTech software for data acquisition and reduction.
	2	HP 5972 MS with capillary direct or split HP 5890 GCs with jet separator interface; each instrument is attached to a Tekmar 3000 liquid sampler (LCS) and Tekmar 2016 sixteen position autosamplers (ALS); both instruments utilize Teklink software interfacing the ALS and HP Chemstation software interfacing the MS for data acquisition; both instruments have a 735 HP/UX Chemserver computer with a Target Operating system for data reduction.
Gas Chromatography	3	HP 5890 GC configured for automatic sampling and equipped with dual Electron Capture Detectors. The GCs are attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3400 GC configured for automatic sampling and equipped with dual Electron Capture Detectors. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	HP 5890 GC configured for automatic sampling and equipped with dual Nitrogen-Phosphorus Detector. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3400 GC configured for automatic sampling and equipped with dual Nitrogen-Phosphorus Detectors. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	2	HP 5890 GCs configured for automatic sampling and equipped with Photoionization and ELCD Detectors. Attached are OI Purge & Trap Autosamplers capable of 16 positions. The GCs are attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	HP 5890 GC configured for automatic sampling and equipped with Photoionization and Flame Detectors. Attached is an OI Purge & Trap Autosampler capable of 16 positions. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.

Table 9-2. List of Laboratory Instruments (Continued, Page 2 of 3)

Analysis Type	Number	Instrument
HPLC	1	Varian 3400 GC configured for automatic sampling and equipped with Photoionization and Flame Detectors. Attached is an OI Purge & Trap Autosampler capable of 16 positions. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3400 GC configured for automatic sampling and equipped with a Photoionization Detector. Attached is a Tekmar LSC2000 Purge & Trap Autosampler capable of 16 positions. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	2	Varian 3400 GC configured for automatic sampling and equipped with a Flame Ionization Detector. One GC is attached to a Waters ExpertEASE Chromatography Data Acquisition System operating under a VAX 3300 computer. The other is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3410 GC configured for automatic sampling and equipped with FID Detectors. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Varian 3400 GC configured for automatic sampling. One GC is equipped with a dual Thermal Selective Detector and FID. The GC is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	PE Sigma 2b GC with a Flame Ionization Detector. The GC is attached to a Waters ExpertEASE Chromatography Data Acquisition System operating under a VAX 3300 computer.
	1	Waters 600E powerline Gradient HPLC Systems equipped with a Fluorescence Detector and is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system. The system is capable of post column derivitization.
	1	Waters 600E powerline Gradient HPLC System equipped with Fluorescence & Diode Array and 484 UV Detectors and post column derivitization; system is attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	2	Waters 600E powerline Gradient HPLC System equipped with a scanning Fluorescence and UV Detector and attached to a personal computer (PC) via a Perkin Elmer interface with the Perkin Elmer (PE) Turbochrome 4.0 chromatography data system.
	1	Waters Milli-Lab Gel Permeation Chromatograph (GPC).
GPC	1	Zymark Benchmate with Waters Gel Permeation Chromatograph (GPC).

Table 9-2. List of Laboratory Instruments (Continued, Page 3 of 3)

Analysis Type	Number	Instrument
Metals	1	Jarrel-Ash 61E Inductively Coupled Plasma (ICP) Emission Simultaneous Spectrophotometer System with automated sampling accessories.
	1	Perkin-Elmer 5500B Inductively Coupled Plasma (ICP) Emission Sequential Spectrophotometer System.
	1	Perkin-Elmer Model 4100ZL Atomic Absorption Spectrophotometer System equipped with Graphite Furnace, Zeeman Background Correction and automated sampling accessories.
	1	Perkin-Elmer 5100 Atomic Absorption Spectrophotometer System equipped with Graphite Furnace, Zeeman Background Correction and automated sampling accessories.
	1	Leeman Model PS200 Cold Vapor Mercury Analyzer with automated sampling accessories.
	2	CEM MSD 2100 Microwave Digestion Systems with automated accessories.
Inorganics	1	Astro Model 2001 System 2 TOC Analyzer.
	1	MCI Model TOX-10 TOX Analyzer.
	1	Dionex DX100 Ion Chromatograph equipped with a AS 40 Automated Sampler, ASRS-I 4 mm Anion Self-Regenerating Suppressor, and AS9-SC Ion Pac Column; instrument uses a Dionex Advance Computer Interface utilizing AI 450 CalPlot software for data acquisition and reduction.
	1	Lachat Model 1200 Autoanalyzer with autosampler.
	2	Orion EA940 and 720A Specific Ion Meters.
	1	Milton Roy Model 301 Visible Spectrophotometers.
	1	Hach Model DR13 Visible Spectrophotometer.
	1	Parr Model 1241 Adiabatic Bomb Calorimeter.
	1	Hach 2100A Turbidimeter.
	2	Hach COD Reactors.
	4	YSI Model 58 DO Meters.
	1	Tecator Soxtec Extraction Unit.
	1	Jenco Electronics, LTD. Model 1671 Conductivity Meter.
	3	Mettler PJ300 (1), Mettler AE240 (1), and Mettler AE163 (1) Analytical Balances.
	8	Ohaus GA200D(1), Ohaus AP1105 Plus(1), Ohaus E4000D(3), Ohaus E400(1), Ohaus C305S(2) Analytical Balances.
	2	American Scientific Products Z-3000-DR Top Loader (1) and SP180 Analytical Balance (1).

Specific calibration requirements for major classes of analytical procedures are described in Section 9. If the calibration requirements of the specified analytical method are more stringent than the procedures described in this QAP, the method procedures will be followed.

9.3.1

**GAS CHROMATOGRAPH/HIGH PRESSURE LIQUID
CHROMATOGRAPH (GC-NONVOLATILES/HPLC)
CALIBRATION**

Single Point Calibration--Single point calibration is a viable alternative to a calibration curve for gas chromatography drinking water methods. (This is not applicable to gas chromatography volatile drinking water methods.) Single point standards are prepared from the secondary standard dilutions. The single point standard is prepared at concentration that produces a response that deviates no more than twenty percent from the sample extract response. The single point calibration is only used when analytes of interest are below the specified reporting limits. If an analyte of interest is detected above the reporting limit, the sample is reanalyzed using a three point standard curve calibration. The procedures for a standard curve calibration are presented below.

Standard Curve Calibration--Initial calibration standard solutions are prepared by serial dilutions of a single stock standard solution to cover the analytical working range of the method. These are either composite standards of more than one analyte or single-analyte solutions. The concentrations are adjusted to take into account the instrumental and method detection limit. A minimum of three initial calibration standard concentrations or the number of standards specified by the method covering the desired working range are prepared and analyzed with a blank. A medium level standard and a blank are analyzed at the beginning of each continuing analytical run. At least one calibration standard at the middle or high range of the curve is analyzed every 10 samples and repeated at the end of the run.

The initial calibration curve is produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. The concentrations of the standards are expressed in terms of the concentration of the standard solution, because the injection volume is constant for standards and samples. QC evaluation criteria for initial calibration, recalibration, and continuing calibrations are as follows:

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1. The initial calibration curve and the subsequent recalibrations possess a minimum of three points and a blank or possess the number of calibration standards specified by the method,
2. The correlation coefficient of the curve is 0.995 or greater,
3. Continuing calibration standard response factors are within 15 percent of the initial calibration for the EPA SW-846 gas chromatography methods, 10 percent for the EPA 600 series gas chromatography methods, 20 percent for drinking water gas chromatography methods, and 10 percent for HPLC methods. Data is not rejected due to an ending standard that fails QC requirements, and
4. The calibration curve brackets the response for all samples.

Corrective actions taken if these calibration QC criteria are not met are listed in Section 13.0.

The concentration (or amount) of the injected sample is obtained by entering the response for the sample into the initial calibration curve equation and determining the sample concentration after all appropriate extract and sample dilution factors have been applied.

9.3.2 GAS CHROMATOGRAPH (GC-VOLATILES) CALIBRATION

Standard Curve Calibration--Calibration standard solutions are prepared as needed by dilutions of several intermediate standard solutions, covering the analytical working range of the method. These are either composite standards of more than one analyte or single-analyte solutions. The concentrations are adjusted to take into account the instrumental and method detection limit. A minimum of three calibration standard concentrations, or the number of standards specified by the method covering the working range are prepared and analyzed with a blank. At least one calibration standard at the middle to high range of the curve is analyzed every 10 samples. GC-volatile methods do not require an ending standard. Calibration is the same as described in Section 9.3.1.

9.3.3 GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) TUNING AND CALIBRATION

GC/MS Tuning--Daily verification of instrument tuning is practiced to ensure the instrument is calibrated and in proper working condition. The GC/MS tune is verified daily with decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis and bromofluorobenzene (BFB) for volatiles analysis. The mass intensity specifications for BFB and DFTPP are contained in Table 9-3.

GC/MS Calibration--Relative response factors for the individual compounds is determined as follows:

$$RF = \frac{A_C Q_{IS}}{A_{IS} Q_C}$$

where: A = integrated area taken from the extracted ion
current profile,
Q = quantity of material,
C = compound, and
IS = internal standard.

An initial calibration with a minimum of three points (or the number of standards per method requirements) is analyzed before samples are analyzed to determine the instrument linearity. The average response factor (RF) is calculated for each compound. The response factors for the System Performance Check Compounds (SPCCs) are ≥ 0.300 except for bromoform which is ≥ 0.250 for EPA 624, EPA 8240, and EPA 8260. The percent relative standard deviation (%RSD) is calculated from the response

Table 9-3. Mass Intensity Specifications for DFTPP and BFB

Key Ions	Ion Abundance Criterion
<u>For DFTPP*</u>	
51	30 to 60 percent of mass 198
68	Less than 2 percent of mass 69
70	Less than 2 percent of mass 69
127	40 to 60 percent of mass 198
197	Less than 1 percent of mass 198
198	Base peak, 100-percent relative abundance
199	5 to 9 percent of mass 198
275	10 to 30 percent of mass 198
365	Greater than 1 percent of mass 198
441	Present but less than mass 443
442	Greater than 40 percent of mass 198
443	17 to 23 percent of mass 442
<u>For BFB*</u>	
50	15 to 40 percent of mass 95
75	30 to 60 percent of mass 95
95	Base peak, 100-percent relative abundance
96	5 to 9 percent of mass 95
173	Less than 2 percent of mass 174
174	Greater than 50 percent of mass 95
175	5 to 9 percent of mass 174
176	Greater than 95 percent but less than 101 percent of mass 174
177	5 to 9 percent of mass 176

*Reference: Test Methods for Evaluating Solid Waste, EPA-SW-846, 3rd Edition, November 1986.

Source: ESE.

factors of each calibration check compound (CCC). Response factors are within 30 percent relative standard deviation for EPA 624, EPA 8240, and EPA 8260. The percent relative standard deviation for the remainder of the compound list is a maximum of 40 percent. For EPA 524.2, the initial calibration is within 20 percent relative standard deviation for all compounds. For EPA 8270, the initial calibration is within 30 percent relative standard deviation for the CCCs. The response factors for the SPCCs are \geq 0.050. For EPA 625, the initial calibration is <35 percent relative standard deviation for all compounds.

A 1-point calibration using a midlevel standard from the initial calibration is used daily for all subsequent analysis, except for Method 524.2 where the analytes are quantitated directly from the calibration curve. For EPA 624, EPA 8240, and EPA 8260, the CCCs are within 25 percent difference of the average response factor of the initial calibration. The SPCCs have the same criteria as the initial calibration. For EPA 524.2, the CCCs are within 30 percent difference of the average response factor of the initial calibration. For EPA 8270, the CCCs and SPCCs have criteria as the initial calibration. For EPA 625, the CCCs are within 20 percent difference of the average response factor of the initial calibration. Corrective actions taken if the QC criteria for calibrations are not met are listed in Section 13.0.

The minimum required internal standards (IS) are chlorobenzene-d5, 1,2-dichloroethane-d4, and 1,4-dichlorobenzene-d4, (in addition, fluorobenzene for 524.2) for volatiles (EPA 624 and 8240); and 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 for semivolatiles (EPA 625 and 8270). A retention time and response check is performed on every internal standard for samples that are analyzed.

9.3.4 GENERAL INORGANIC AND ORGANIC PARAMETERS CALIBRATION

Standard Curve Calibration--This section applies to those inorganic and organic analyses procedures [ion chromatography, colorimetric, spectrophotometric, ultraviolet (UV)]

absorption, turbidimetric] that use a standard curve for calibration [except total organic carbon (TOC), chemical oxygen demand (COD), infrared (IR), and potentiometric].

Working standard solutions are prepared by serial dilution of a single-stock standard to bracket the analytical working range of the method. Working standard solutions are either composite standards of more than one analyte or single-analyte solutions. The standard concentrations are adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. A minimum of three standard concentrations, or the number of standards specified by the method, covering the working range are prepared and analyzed with a blank. A continuing working standard and a blank are analyzed, at a minimum, at the beginning of every analytical run; and at least one midlevel standard, which is the continuing calibration verification (CCV) standard, is reanalyzed at minimum intervals of every 20 samples and at the end of the run to check for constant instrument response.

The preparation of calibration standards is verified by the analysis of the ICV solution. The initial calibration verification (ICV) is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. Typically, the standards are from the same supplier, but from a different lot. Certificates of Analysis are available for all standards.

The working curve is produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. QC evaluation criteria for working curves are as follows:

1. The working curve possesses a minimum of three points, or the number of standards specified by the method, and a blank;
2. The correlation coefficient of the line is 0.995 or greater;
3. The response for the CCV analyzed at minimum intervals of every 20 samples during the run and at the end of the run is within 20 percent of true value
4. The ICV is within 20 percent of the element's true value; and
5. The calibration curve brackets the response for all samples.

Corrective action procedures taken if these QC evaluation criteria are not met are provided in Section 13.0. The sample concentration is obtained by entering the response for the sample into the working curve equation and determining the sample concentration after all appropriate extract and sample dilution factors have been applied.

9.3.5 TRACE METALS ANALYSIS CALIBRATION

Atomic Absorption Spectroscopy (AAS) Standard Curve Calibration--Working standard solutions are prepared to include the analytical working range of the method; these solutions are either composite standards of more than one metal or single-metal solutions. The standard concentrations are adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. A minimum of three standard concentrations, or the number of standards specified by the method, covering the working range are prepared and analyzed with a blank. The calibration standards and the blank are analyzed at the beginning of every analytical run, and at least one midlevel standard is analyzed at minimum intervals of every 20 samples during the run and at the end of the run to check for constant instrument response.

The calibration is verified by the analysis of the ICV solution. The ICV is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. Typically an EPA or NIST reference is used as the ICV and is prepared according to the supplier's instructions.

The working curve is produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run. QC evaluation criteria for working curves are as follows:

1. The working curve possesses a minimum of three points, or the number of standards specified by the method, and a blank;
2. The correlation coefficient of the line is 0.995 or greater;

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3. The response for the midlevel standard, analyzed at minimum intervals of every 20 samples during the run and at the end of the run, is within 20 percent of true value;
4. The ICV is within 10 percent of the element's true value; and
5. The calibration curve brackets the response for all samples.

Refer to Section 13.0 for the corrective action procedures taken if these QC evaluation criteria for calibration are not met. The concentration of a trace metal in the sample is obtained by entering the response for the sample into the working calibration curve equation and determining the metal concentration in the digestate. The value is corrected by the appropriate digestate volume, sample size, applicable dilution factor, and moisture content (for soils) to generate a final sample concentration.

Inductively Coupled Argon Plasma (ICAP) Single Point Calibration--This procedure uses a single standard concentration for each element to obtain an instrument response (emission counts) and is analyzed in every analytical run. A second single point, emission counts obtained when aspirating a blank solution (undigested, acidified DI water), is used in conjunction with the standard to calibrate the instrument in concentration units.

The calibration is verified by the analysis of an ICV solution, which is an independent standard prepared from different stock solutions than those used to prepare the calibration standards. The elemental concentrations of the calibration verification solution must be within the calibration range of the instrument and at concentrations other than those used for instrument calibration.

A multi-element interference check solution (ICS) and a method blank (acidified DI water that is carried through the digestion process) are analyzed each day prior to analyzing the samples. The ICS is used to verify the correction of spectroscopic interference caused by emissions adjacent to analyte emission lines.

The CCV solution is analyzed at minimum intervals of every 20 samples during the run and at the end of the run to document constant instrument response. This solution is in the midrange of each element present in the calibration standards. This solution may be prepared by dilution of an aliquot of the calibration standard or prepared as a separate solution in a manner analogous to the calibration standard preparation procedure.

QC evaluation criteria for the instrument calibration standard are as follows:

1. A calibration standard and a calibration blank are used;
2. All the values for the ICV are within 10 percent of each element's true value;
3. Values for the ICS are 20 percent of each element's true value; and
4. The measured concentrations of the elements in the CCV solution, for which calibration was performed, are within 10 percent of their respective true values.

Corrective action procedures if these QC evaluation criteria are not met are provided in Section 13.0.

9.3.6 GRAVIMETRIC METHODS CALIBRATION

Two general types of analytical balances are used at ESE: (1) the more sensitive microanalytical balance and (2) the top-loading balance. The calibration of the microanalytical balances is verified daily by weighing the following Class S and NIST-certified weights [in grams (g)]:

<u>Weight (g)</u>	<u>Tolerance Limits</u>
0.1	± 0.0005
0.5	± 0.0005
1.0	± 0.0005
3.0	± 0.0005

The calibration of the top loading balances are verified daily by weighing the following Class S and NIST-certified weights:

Weight (g)

Tolerance Limits

5	± 0.02
20	± 0.05
50	± 0.05

The calibration results are recorded in the appropriate balance logbook. If these criteria are not met, the weight may be reweighed. If the criteria are not met for the second weighing, the balance is taken out of service and repaired. Two sets of Class S weights are available in-house. Qualified service personnel calibrate the analytical balances semiannually, utilizing Class S and NIST certified weights. The semiannual calibration is documented by a tag on the instrument. Service calibration records are kept on file in the laboratory.

9.3.7 TITRIMETRIC METHODS CALIBRATION

In all cases, prepared standards are used to calibrate the titrant and back titrant. Preparation of these materials is described in Standard Methods or other method manuals. Known solutions of the parameter to be analyzed are prepared and analyzed to verify titrant standardization and the analyst's ability to discern the endpoint.

9.3.8 TOC CALIBRATION

The TOC analyzer is calibrated with a prepared standard using a single-point calibration. The standard is analyzed before beginning every analytical run. The continuing calibration verification standard (using mid- to high-level standard) is analyzed every 10 samples and at the end of the run, and the response must be within ± 15 percent of true value.

9.3.9 COD CALIBRATION

Prepared standards are used to verify the 0- and 500-mg/L readings with the standard curve. The standard curve is developed by Hach Chemical Company for COD on a spectrophotometer using prepared sample vials. The 500-milligrams-per-liter (mg/L) standard must be within 5 percent.

9.3.10 BOD CALIBRATION

The oxygen probe is calibrated daily according to the manufacturer's air calibration procedure. The temperature of the incubator used for the BOD analysis will be read and recorded daily when in use.

9.3.11 TOTAL ORGANIC HALIDES (TOX) CALIBRATION

The TOX analyzer is calibrated with a prepared standard using a 3-point calibration. The linearity of the calibration is verified with a low-level and high-level standard to bracket the sample concentration. The linearity checks must be within 5 percent. The continuing calibration verification standard (using mid- to high-level standard) is analyzed every 10 samples, and the response must be within ± 15 percent of true value.

9.3.12 pH CALIBRATION

The pH meter is calibrated with three buffer solutions at pH 4, pH 7, and pH 10 prior to use. The pH meter temperature selector is set to ambient temperature. The probe is placed on the pH 7 buffer; and the calibration switch is adjusted until it reads 7.00 units. The initial pH value and the buffer adjusted value should be recorded. The procedure is repeated with the pH 4 and pH 10 buffer solutions. The pH of the pH10 buffer should be 10 ± 0.05 units; if not, the pH probe and internal solution is checked and the calibration procedure is repeated.

9.3.13 SPECIFIC CONDUCTIVITY CALIBRATION

The instrument is calibrated with 0.01 M and 0.10 M KCL solutions. The conductivity reading of the 0.01 M KCL must be 1,413 umhos $\pm 15\%$; the 0.10 M KCL must be 12,900 units $\pm 15\%$. If the calibration standards are outside the acceptance criteria, new standards are prepared and the instrument is recalibrated.

9.3.14 PENSKY-MARTENS CLOSE-CUP TESTER CALIBRATION

The ignitibility of the p-xylene standard is determined prior to use of the Pensky-Martens Close-Cup Tester. The standard should ignite at $27.2 \pm 1^\circ\text{C}$. If not, the condition and

operation of the apparatus is checked, especially the tightness of the lid, the action of the shutter, and the position of the test flame. After adjustment, the test is repeated with the p-xylene standard. The barometric pressure is read and recorded at the time of analysis.

9.3.15 DISSOLVED OXYGEN CALIBRATION

The dissolved oxygen probe is calibrated daily or prior to use in saturated air by moving the calibration knob such that the reading is at the appropriate saturation value indicated on the instrument. The temperature is read and recorded at the time of analysis.

9.4 STANDARDIZATION OF TITRATION SOLUTIONS

All titrants used in the laboratory are standardized against a primary standard. This ensures that the normality of the standard being used is at the correct level. Table 9-4 lists the solutions that require standardization, the standards used, and the frequency of standardization.

Table 9-4. Standardization of Titrating Solutions

Solutions Req.	Primary Standard Source	Frequency of Standardization
Chloride: Silver nitrate	Sodium chloride	Every run
Alkalinity: Sulfuric acid	Sodium carbonate	Every run
Sulfite: Potassium iodide-iodate	Sulfamic acid	Every run
Hardness: EDTA	Calcium carbonate	Every run

Source: ESE.

10.0 PREVENTIVE MAINTENANCE

To minimize the occurrence of instrument failure and other system malfunctions, a preventive maintenance program for laboratory instruments is implemented. Routine maintenance is performed as needed, depending on how often the instrument is used. Since some parts of the instrument are utilized more than others, replacement for these parts is required more frequently. These wearable or expendable parts are monitored during analysis for optimum performance and kept in supply in the event of instrument failure. Major instruments in the laboratory are covered by service contracts or agreements provided by various vendors.

10.1 DOCUMENTATION

All maintenance performed on the instruments is documented in each instrument's maintenance logbook, which is kept with the instrument. The date, initials of the analyst performing the maintenance, and the type of maintenance performed are recorded in the maintenance logbook. Receipts from the routine maintenance performed by the service representative are filed in the laboratory. Preventive maintenance for each major piece of laboratory equipment is listed in Table 10-1.

10.2 CONTINGENCY PLAN

In the event of instrument failure, every effort is made to analyze samples within holding times by alternate means. If ESE Peoria's additional instrumentation is insufficient to handle the affected samples, efforts are made to secure the same or equivalent analyses by an appropriately certified or validated laboratory. After contact with an alternate laboratory, the Project Manager is advised of any required changes in methodology or sample location; the Project Manager then notifies the appropriate state/government agency and the client of project modifications. Procedures concerning laboratory custody of samples is found in Section 7.4.

Table 10-1. Preventive Maintenance

Instrument	Activity	Frequency
Gel-Permeation	Replace sample/air syringe	As needed
	Check solvent flow	Daily
	Clean injectors	As needed
	Clean/replace guard column frits	As needed
	Change GPC columns	As needed
	Clean detector	As needed
Gas Chromatographs	Change septums	As needed
	Check carrier gas	Daily
	Change carrier gas	As needed (when pressure falls below 500 psi)
	Cut off edge of a capillary column	As needed
	Replace oxygen traps used in the gas lines	As needed
	Clean detectors	As needed
	Replenish detectors	As needed
	Clean detectors	Daily or as needed
	Check system for gas leaks	As needed
	Clean injection ports	Weekly or as needed
High Performance Liquid Chromatographs	Check piston seals	Weekly, replace as needed
	Check, replace or rebuild the check valves	Weekly (replace/rebuild as needed)
	Clean detector flow cell	As needed
	Check pumps	Daily
	Replace guard column frits	As needed
	Clean detectors	As needed
	Degassed and leak checked	Daily
	System/air pressure	Daily
	Auto-injector syringes	Daily
Gas Chromatograph/Mass Spectrometer	Clean source and system	As needed
	Cut off ends of capillary columns	As needed
	Change columns	As needed
	Change injection point liners	As needed
	Change pump oil	As needed
	Check flow level	As needed
	Routine maintenance performed by the manufacturer	Annually

Table 10-1. Preventive Maintenance (Continued, Page 2 of 3)

Instrument	Activity	Frequency
Atomic Absorption Spectrophotometers (Furnace and Cold Vapor)	Clean furnace windows	Daily
	Check plumbing connections	Daily
	Change graphite tubes	As needed
	Clean sample cells	Daily
	Check gases	Daily
	Check optics and routine maintenance by the manufacturer	Annually (on contract)
	Change graphite contact rings	As needed
Inductively Coupled Plasma (ICAP)	Routine maintenance performed by the manufacturer	Annually (on contract)
	Check and clean the torch, nebulizer, and O rings	As needed
	Check tubing	As needed
Cold Vapor Analyzer	Clean adsorption cell	Daily
	Clean gas/liquid separator	Daily
	Replace pump tubing	Weekly
	Change drying column	Weekly
Autoanalyzers	Clean or replace tubing	As needed
	Check tubing	Daily
	Check and clean optics	As needed
	Clean flow cell	As needed
	Replace the lamp	As needed
Colorimeter/Turbidimeters	Check optics	Daily
	Check light source	As needed
Spectrophotometer	Calibrate wavelength	Semiannually
	Replace lamps	As needed
	Replace phototubes	As needed
TOX Analyzer	Clean electrodes	Daily
	Replace all solutions	Daily
	Clean absorber module and the furnace unit	As needed
	Clean sampler boat	As needed
	Check gases and tubing	Daily
	Rebuild agar bridge	As needed
TOC Analyzer	Check gases and tubing	Daily
	Change pump tubes	As needed
	Flush system	After each use

Table 10-1. Preventive Maintenance (Continued, Page 3 of 3)

Instrument	Activity	Frequency
Ion-analyzers/Conductivity	Check probe	Daily
	Change probe solution	As needed
Ion Chromatograph	Check system for leaks	Weekly
	Check line pressure and piston seals	Weekly
	Clean cell electrodes	Monthly
	Clean injection loops	As needed
	Change columns	As needed
	Replace tubing in the sample path	As needed
Turbidimeter	Clean the instrument	Daily
Analytical Balances	Clean the balance	Daily
	Check alignment and balance	Daily
	Routine maintenance and calibration performed by the manufacturer	Semiannually
Ovens: TS, TSS, TDS	Check temperature	Daily
	Calibrate thermometers	Annually
Refrigerators/Freezers	Check temperature	Daily
	Calibrate thermometers	Annually
BOD Incubator	Check temperature	Daily
	Calibrate thermometers	Annually

Note: TDS = total dissolved solids. TS = total solids. TSS = total suspended solids.

11.0 QC CHECKS, ROUTINES TO ASSESS PRECISION AND ACCURACY, AND CALCULATION OF METHOD DETECTION LIMITS

11.1 INTERNAL QC CHECKS

Analytical QC procedures are those steps taken by the laboratory in day-to-day activities to achieve the desired accuracy, precision, reliability, and comparability of analytical data. Each Department Manager is responsible for overseeing the performance of the analysis in accordance with the defined quality control practices outlined in this CQAP.

For all analyses performed by ESE, the QC checks described in this section are mandatory unless alternate procedures are given in a specific project QA Plan or otherwise agreed upon by the Laboratory Manager and the Project Manager. Table 11-1 summarizes minimum QC sample requirements. If method QC requirements are more stringent than those listed in Table 11-1, the method requirements are followed. Sections 5.0 and 9.0 contain QC evaluation criteria for laboratory methods and calibrations. Section 11.2 describes precision and accuracy calculations used for control samples. Laboratory Department Managers are responsible for reviewing QC criteria for each method performed by their department. Permanent changes to the acceptance criteria are approved by the Department Managers, Operation Managers, and QA/QC Coordinator and are incorporated into this document in accordance with Section 3.3. Project-specific revisions are documented in a specific project QA Plan.

For QC purposes, a Sample Delivery Group (SDG) is used to identify a group of samples to be received by the laboratory from a client. The SDG is a set of twenty or fewer environmental samples by matrix (e.g. soil, water, etc.) received by the laboratory from a client over a period of up to fourteen calendar days or seven calendar days if a fourteen day turnaround time is requested. (Data from all samples in a SDG are due on the same date.) If a SDG is not indicated by the client, the number of samples extracted and/or

Table 11-1. Minimum QC Sample Requirements

<u>QC Sample</u>	<u>CLASS™ Code</u>	<u>Frequency</u>	<u>Analysis</u>
Method Blank	MB	Daily or 1 per 20 samples or SDG	All analyses
Standard Spike / Laboratory Control Sample	SP	Daily or 1 per 20 samples or SDG	All analyses except (a)
Sample Matrix Spike**	SPM1	Daily or 1 per 20 samples or SDG (b)	All analyses except (a)
Sample Matrix Spike Duplicate	SPM2	Daily or 1 per 20 samples or SDG	All analyses except (a)
Surrogate***	SUR	All samples (organics only)	Required for all organic samples and standards, when required
Replicate	RP	Daily or SDG	For miscellaneous inorganic parameters (a)
Analytical Spike	SPX	10% of samples or as specified by the method	Required for GFAA and CVAA methods only
Serial Dilution	SD	If SPM fails acceptance criteria only	Required for ICAP only

- (a) Miscellaneous inorganic parameters including: conductivity, pH, residues, DO, % moisture, turbidity, etc.
- (b) TCLP, 5% or 1 per waste type, whichever is greater. Sample Matrix Spike Duplicate not required for this analysis.

SDG Sample Delivery Group

- * Standard Spike (QC Check Standard) is a spike into a blank matrix which is carried through sample preparation, sample digestion, or extraction to sample analysis. The blank matrix is a reagent blank for aqueous and soil samples. This spike is also called a QC Check Standard, because the standards used to prepare the spiking solution are from a different source than those used for the calibration standards.
- ** Sample Matrix Spike is a spike into a sample matrix which is carried through sample preparation, sample digestion, or extraction to sample analysis.
- *** Surrogates are required for all organic methods as appropriate.

prepared for instrumental analysis as one group in one 24-hour period constitute an extraction group. The number and type of QC samples specified in Section 11.0 apply to either a SDG or an extraction group, if a SDG is not specified. For example, a group of samples that is extracted on the same day and (if required) undergoes concentration and cleanup procedures on subsequent days are considered one extraction sample group for QC purposes. For analyses where no sample extraction or preparation is required, the number of samples that can be analyzed as one set during a 24-hour period determines the number of samples per sample group for QC purposes. The number and type of QC samples specified in Section 11.0 also apply to this group of samples.

When required, as for a specific project, the Department Manager may insert into a current sample batch either spiked sample or sample duplicate results of a previously analyzed sample for QC purposes (with all previous batch references documented in the current batch folder). The Department Manager reviews the results of the previous sample batch to ensure that the analysis meets QC criteria for the current project.

Blind QC check samples are samples of known composition by the QA/QC Coordinator, USEPA, etc., but of unknown composition to the analyst. Blind QC check samples from the USEPA are analyzed by the laboratory semiannually to evaluate the laboratory's analytical performance. If the blind QC check sample data are not acceptable, a corrective action summary report is written and submitted to appropriate states and agencies for certification requirements.

A sample matrix spike (SPM1) is defined as an environmental sample to which known concentrations of control analytes have been added. In addition, if enough sample is present, the sample is split into a duplicate, known as a matrix spike duplicate (SPM2). Sample matrix spikes are included in batch QC for all analyses except miscellaneous inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, and turbidity. Results of the sample, and SPM1/2 pair are used to generate

recoveries. This data is used to assess the accuracy of the analytical procedure (percent recovery) and indicate and matrix interferences. SPM1/2 results are also used to assess the precision (relative percent difference) of the analytical procedure. Selection of the sample to be spit and spiked is specified by the client or the laboratory. Results are reported on a per batch basis.

Control spikes (standard matrix spikes (SP) or QC check standards) are placed into blank matrices for all analyses except miscellaneous inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, and turbidity. This spike is used for method control and verifies the calibration standards, if an ICV is not analyzed. A sample replicate is prepared and analyzed for inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, and turbidity. The relative percent difference between the sample and the replicate is used to assess analytical precision.

It is ESE's policy to control sample analyses with QC criteria that are under the control of the technicians and analysts utilizing the analytical procedure. Therefore, emphasis is placed on calibration, method blanks, and standard matrix spike results. When these QC sample results are within criteria, acceptable method performance is documented. Sample matrix spikes are reported and evaluated for precision and accuracy, but not necessarily used for method control. A sample matrix spike that has recoveries outside of QC criteria is evaluated against other available QC data, within the batch, to determine if the method is in control and if sample flagging is warranted. The failure of a sample matrix spike to achieve acceptable QC criteria when a standard matrix spike in the same batch has acceptable recoveries, indicates whether or not the sample matrix interferes with the quantitation of the target analytes. Cases where poor precision or erratic recoveries are seen indicate that the analysis method selected for the samples may not be appropriate for that matrix type, not that the method is out of control.

Precision and spike recovery checks are discussed in further detail in Section 11.2.

11.1.1 GC/MS MINIMUM QC

For GC/MS analyses, the following minimum QC checks apply, except for CLP SOW:

1. All samples spiked with surrogate.
2. At least 5 percent spikes in a sample matrix (SPM1) with selected analytes and surrogates are analyzed.
3. At least 5 percent duplicate spikes in a sample matrix (SPM2) with selected analytes and surrogates are analyzed.
4. At least 5 percent QC standard spikes (SP) in a blank matrix with selected analytes and surrogates are analyzed.
5. At least 5 percent method blanks spiked with surrogates are analyzed.
6. An initial calibration with a minimum of three points (or the number of standards per method requirements) is analyzed before samples are analyzed. Response factors for the Calibration Check Compounds (CCCs) are within 30 percent relative standard deviation for EPA 624, EPA 8240, and EPA 8260. The response factors for the System Performance Check Compounds (SPCCs) are ≥ 0.300 except for bromoform which is ≥ 0.250 . The percent relative standard deviation for the remainder of the compound lists is a maximum of 40 percent. For EPA 524.2, the initial calibration is within 20 percent relative standard deviation for all compounds. For EPA 8270, the initial calibration is within 30 percent relative standard deviation for the CCCs. The response factors for the SPCCs are ≥ 0.050 . For EPA 625, the initial calibration is < 35 percent relative standard deviation for all compounds.
7. Instrument tuning protocols are performed and are within criteria (listed in Section 9) prior to analysis.
8. Continuing calibration standard is analyzed at a frequency of 5 percent or at the beginning of a daily continuing analytical sequence. For EPA 624, EPA 8240, and EPA 8260, the CCCs are within 25 percent difference of the average response factor of the initial calibration. The SPCCs have the same criteria as the initial calibration. For EPA 524.2, the CCCs are within 30 percent difference of the average response factor of the initial calibration. For

EPA 8270, the CCCs and SPCCs have the same criteria as the initial calibration. For EPA 625, the CCCs are within 20 percent difference of the average response factor of the initial calibration.

9. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.
10. Samples are within the concentration range of the standards.

11.1.2 GC AND HPLC MINIMUM QC

For GC-nonvolatiles, GC-volatiles, and HPLC analyses the following minimum requirements apply, except for CLP SOW:

1. All samples spiked with surrogate(s), if specified by the method.
2. At least 5 percent spikes in a sample matrix (SPM1) with selected analytes and surrogate(s) (if applicable) are analyzed.
3. At least 5 percent duplicate spikes in a sample matrix (SPM2) with selected analytes and surrogate(s) (if applicable) are analyzed.
4. At least 5 percent QC standard spikes (SP) in a blank matrix with selected analytes and surrogate(s) (if applicable) are analyzed.
5. At least 5 percent method blanks spiked with surrogate(s) (if applicable) are analyzed.
6. A minimum of three standards or the number of standards specified by the method are analyzed as a standard curve except for non-volatile drinking water methods where single point calibration, as described in Section 9, is applicable.
7. Correlation coefficient of the standard curve is equal to or greater than 0.995.
8. Samples are within the concentration range of the standards.
9. Midlevel calibration standards are repeated at minimum intervals of every 10 samples and at the end of a run (except GC-volatiles), and response factors are within 15 percent of the initial calibration for the EPA SW-846 gas chromatography methods, 10 percent for the EPA 600 series gas chromatography methods, 20 percent for drinking water gas chromatography

methods, and 10 percent for HPLC methods. Data is not rejected due to an ending standard that fails QC requirements. GC-volatile methods do not require an ending standard; midlevel calibration standards are analyzed.

10. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.

11.1.3 TRACE METALS--ATOMIC ABSORPTION AND ICAP SPECTROSCOPY MINIMUM QC

For each batch of samples analyzed by AAS or ICAP, the following QC checks apply, except for CLP SOW:

1. At least 5 percent spikes in a sample matrix (SPM1) with selected elements are analyzed.
2. At least 5 percent duplicate spikes in a sample matrix (SPM2) with selected elements are analyzed.
3. At least 5 percent QC standard spikes (SP) in a blank matrix with selected elements are analyzed.
4. At least 5 percent method blanks are analyzed.
5. A minimum of three standards or the number of standards specified by the method are analyzed as a standard curve.
6. Correlation coefficient of the standard curve is equal to or greater than 0.995.
7. Samples are within the concentration range of the standards (or of the ICAP instrument).
8. Midlevel calibration standards are repeated at minimum intervals of every 20 samples and at the end of a run. Response of the elements are within 20 percent of the true value for CVAA and GFAA (10 percent for ICAP).
9. Detection limits for each element are determined and checked to ensure they meet reporting limit requirements specified for the project.

11.1.4 MISCELLANEOUS METHODS MINIMUM QC

For each batch of samples analyzed by ion chromatographic, colorimetric, spectrophotometric, IR, UV absorption, and titrimetric methods (except for additional miscellaneous inorganic methods such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, turbidity), the following QC checks apply:

1. At least 5 percent QC standard spikes (SP) in a blank matrix are analyzed.
2. At least 5 percent spikes in a sample matrix (SPM1) are analyzed.
3. At least 5 percent duplicate control spikes in a sample matrix (SPM2) are analyzed.
4. At least 5 percent method blanks are analyzed.
5. A minimum of three standards or the number of standards specified by the method are analyzed as a standard curve.
6. Correlation coefficient of the standard curve is equal to or greater than 0.995.
7. Samples are within the concentration range of the standards.
8. Midlevel calibration standards are repeated at minimum intervals of every 20 samples and at the end of a run. Responses of the standards are within 20 percent of the true value.
9. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.

For each batch of samples analyzed for additional inorganic parameters such as pH, residues, dissolved oxygen, % moisture/solids, conductivity, turbidity, the following QC checks apply:

1. At least 5 percent sample replicates are analyzed.
2. At least 5 percent method blanks are analyzed.
3. Continuing calibration standards, if applicable, are analyzed at a frequency of 5 percent.
4. Detection limits for each parameter are determined and checked to ensure they meet reporting limit requirements specified for the project.

11.2 ROUTINE METHODS USED TO ASSESS PRECISION AND ACCURACY

11.2.1 PRECISION

Precision is the degree of mutual agreement among individual measurements repeatedly performed utilizing the same test procedure and conditions. Precision is assessed for applicable parameters by calculating the RPD of two duplicate spike samples as follows:

$$RPD = \frac{|R_1 - R_2|}{(R_1 + R_2)/2} \times 100$$

where: R_1 and R_2 = concentration of Replicate Spikes 1 and 2,
respectively.

This calculated RPD value is compared to the criteria specified in Section 5 of the CQAP. Additionally, the spike levels used to determine the precision targets are listed in Section 5.

11.2.2 ACCURACY

Accuracy is the degree of agreement between a sample's target value (true or expected concentration) and the actual measured value. Accuracy for this project is measured by calculating the percent recovery (R) of known levels of spike compounds into appropriate sample matrices. Percent recovery is calculated as follows:

$$R = \frac{100 \times [(Spike Sample Conc.) (Sample + Spike Vol.) - (Sample Vol.) (Sample Conc.)]}{(Spike Conc.) (Spike Volume)}$$

The following equation is an example calculation:

1 mL of spike with concentration of 100 ppb
10 mL of sample with concentration of 10 ppb
spiked sample concentration of 20 ppb

$$= 100 \times \frac{(20)(11) - (10)(10)}{(1)(10)} = 100 \times \frac{120}{100} = 120 \text{ percent}$$

Each calculated R value is compared to accuracy criteria listed in Section 5. The accuracy ranges provided in Section 5 are based on the mean accuracy measured or expected, as from method criteria, for each parameter plus or minus three standard deviations of the mean. The spike levels used to determine the accuracy targets are listed in Section 5. If RPD or R values for standard spikes within a batch do not meet acceptance criteria specified in Section 5, the batched samples are re-analyzed or sample results are flagged appropriately. If nonconformances occur, the Department Manager or designee is notified and necessary corrective action taken. Proper corrective action procedures are described in Section 13.

11.2.3 EVALUATION OF CONTROL CHARTS

Control charts are graphical plots of analysis results that illustrate statistical control by monitoring trends in a measurement process through time or sequence of analysis. By monitoring the measurement process, control limits are generated to demonstrate that the method is statistically in control. It is improbable that a point could lie outside the limits on a control chart while the system remains in a state of control.

Analysts have the ability, through the ESE Laboratory Information Management System CLASS™ to generate control limits using historical ESE data. If sufficient in-house data is unavailable, control limits are derived from published USEPA method data, if available. Control limits are updated yearly or as needed using historical ESE data.

The formulas used to establish and maintain control limits for laboratory standard spike QC samples are as follows:

$$\begin{aligned} \text{UCL}_{-x} &= \bar{X} + 3\text{SD} \\ \text{UWL}_{-x} &= \bar{X} + 2\text{SD} \\ \text{LWL}_{-x} &= \bar{X} - 2\text{SD} \end{aligned}$$

$$LCL_{-x} = \bar{X} - 3SD$$

where: \bar{X} = Mean of the recoveries of the laboratory spikes,
 SD = Standard deviation of the mean,
 UCL = Upper control limit,
 UWL = Upper warning limit,
 LWL = Lower warning limit, and
 LCL = Lower control limit.

All control limits are specifically tabulated according to matrix and QC type.

An analysis is considered out of control when any one of the following situations exist:

1. One point plots outside the control limits,
2. Eight consecutive points plot on the same side of the mean,
3. A systematic pattern is evident,
4. Three consecutive points plot within the control limits but outside the warning limits.

The occurrence of any of these events is investigated and corrective actions are taken as required to return the system to a state of statistical control. Corrective actions are documented using the appropriate corrective action form, Section 13.

11.3 METHOD DETECTION LIMITS AND PRACTICAL QUANTITATION LIMITS

11.3.1 METHOD DETECTION LIMITS (MDLs)

The detection limit of a method is the lowest sample concentration which is reliably recovered and measured in the sample matrix with a low background level. To determine absolute MDLs, statistically based procedures are available from EPA methods.

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Minimally, MDL studies are performed annually for methods routinely used by the laboratory.

The detection limit is defined (40 CFR, Part 136 Appendix B) as follows for all measurements:

$$MDL = t_{(n-1, 1-\alpha, = 0.99)} \times S$$

where:

MDL = Method detection limit,

S = Standard deviation of the replicate analyses, and

$t_{(n-1, 1-\alpha, = 0.99)}$ = Students t-value appropriate to a 99-percent confidence level and a standard deviation estimate with n-1 degrees of freedom.

Instrument Detection Limits (IDL) are calculated similarly to the MDLs. Instead of the detection limit study being performed in a sample matrix that has gone through the appropriate extraction or digestion procedure, IDLs are generated by repetitively analyzing standard matrix spikes in the same manner discussed in the 40 CFR Part 136 Appendix B.

11.3.2 PRACTICAL QUANTITATION LIMIT (PQL)

The PQL is the lowest concentration of an analyte that can be reliably achieved within a specified degree of precision and accuracy throughout routine laboratory conditions. The PQL is defined as approximately three to five times the Method Detection Limit. The PQL or reporting limit may be modified to meet clients' specifications.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

12.1 DATA REDUCTION

Data transfer and reduction are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and, in the case of data reduction, that accepted statistical techniques are used. ESE uses the company developed Laboratory Information Management System, CLASS™, for all projects.

If applicable, example calculations are included with the summarized data to facilitate review. The entry of input data and calculations are checked and the signature/initials of the analyst or individual entering the data and reviewer(s) accompany all data transferred (with and without data reduction). All final analysis results are calculated according to the referenced methods specified in Section 5.

For routine analyses performed at the Peoria Laboratory, sample response data is entered into CLASS™ by the analyst or other designated individual(s). The computer calculates the following:

1. Linear, quadratic, or logarithmic regression line for standards,
2. Coefficients of variation for replicates,
3. Spiked recoveries, and
4. Sample concentrations.

Linear or quadratic equations are used to calculate final data for laboratory analyses requiring a calibration curve:

Linear: $\text{Concentration} = \text{Intercept} + M (\text{Response})$

Quadratic: $\text{Concentration} = \text{Intercept} + M (\text{Response}) + M2 (\text{Response})^2$

The equation used to calculate final data is dependent on the linearity of the standard curve and methodology of analysis.

Purgeable organics by GC/MS are calculated as follows:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_{sa})(Q_{is})}{(RF)(A_{is})(PV)}$$

where: A_{sa} = area from the extracted ion profile of the primary characteristic ion for the target analyte in the sample,

Q_{is} = quantity of the internal standard [nanograms (ng)],

RF = response factor (see Section 8),

A_{is} = area from the extracted ion profile of the primary characteristic ion of the internal standard in the sample, and

PV = purge volume (mL).

Semivolatile organics by GC/MS are calculated as follows:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_{sa})(Q_{is})}{(A_{is})(RF)} \times \frac{1}{FE} \times \frac{1}{\text{volume}} \times DF$$

where: A_{sa} = area from the extracted ion profile of the primary characteristic ion for the target analyte in the sample,

A_{is} = area from the extracted ion profile of the primary characteristic ion of the internal standard in the sample,

Q_{is} = quantity of the internal standard (ng),

RF = response factor (see Section 8),

FE = fraction extract analyzed = $\frac{\text{Volume injected } (\mu\text{L})}{\text{Extract volume } (\mu\text{L})}$,

volume = volume of extracted sample (mL), and

DF = dilution factor = $\frac{\text{final extract volume for injection (mL)}}{\text{extract volume prior to dilution (mL)}}$.

The final data for GC/MS semivolatiles and volatiles analyses are calculated by the computer data acquisition system attached to each mass spectrometer.

QC acceptance criteria (Section 5) for the relative percent difference of replicate spike recoveries and the range of acceptable spike recoveries are electronically stored in the computer data management files for each STORET number/method code combination. If the samples in a batch (sample group) do not pass all the QC checks (Section 11), the results reported for all samples processed in the same sample group are considered as suspect and flagged if appropriate; analyses may need to be repeated.

Completed batch folders are stored in a secured central location arranged by departments and numerically by batch number. Chromatograms, copies of parameter notebooks, and all other pertinent raw data and other documentation are stored in the batch folders.

Once the data set is complete for each sampling effort, the Project Manager organizes the information for final report format, according to project requirements. The Project Manager is responsible for final QC review and release of the data.

12.1.1 THE DOCUMENTATION RECORDS

All manual documentation of raw data is done in notebooks or appropriate forms. All notebooks used have consecutively numbered pages. All notebook entries are made in indelible ink. Any blank portions of data forms or notebook pages are lined out with black ink and initialed by the analyst.

12.1.1.1 GC/HPLC

Extraction Logbook--An extraction logbook copy, filled out by the analyst performing the sample extraction, accompanies each lot of samples throughout analysis. This sheet includes at least the following data:

1. Project name,
2. Extractor's initials,

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3. Type of sample matrix,
4. Field group name,
5. Sample numbers,
6. Date extracted,
7. Analytical method,
8. Initial volume or wet weight of sample extracted,
9. Initial pH (water sample),
10. Extracting solvent,
11. Final volume/solvent,
12. Extract box identification,
13. Date of cleanup (if required),
14. Notes and comments affecting the extraction procedure, and
15. Surrogate/spike preparation reference number and spike volume.

After extraction is complete, the extraction logbook copies accompany the sample arrival notice to the instrumental analyst. The extracted samples are refrigerated and stored in boxes, in a central location, until the required analysis. The box number is referenced on the extraction logbook copy. Each extract vial is properly labeled and include the following information:

1. Project name,
2. Field group name,
3. Sample number,
4. Analyte group and matrix,
5. Date extracted, and
6. Extraction logbook reference number.

Instrument Logbooks--During analysis, the following information is recorded in an instrument notebook:

1. A log of the types of analyses run on the instrument, to include:

- a. Column/instrument conditions and temperature zones,
 - b. Sample numbers or other identification of samples,
 - c. Reference to a method or analyte group describing the analysis,
 - d. Sequence date and analyst initials,
 - e. Detector used [e.g., flame ionization detector (FID)] (on cover), and
 - f. Detector conditions.
2. Service records are kept in a separate maintenance log.

Chromatograms--The analyst will include the following information on the chromatogram (if not automatically printed):

1. Date and time of analysis,
2. Analyst identification,
3. Instrument used,
4. Field group name,
5. Sample number and other identification for each chromatogram, and
6. Concentration/dilution factor for each sample (not for GPC).

After the analysis and data reduction are complete, the chromatograms are stored in the batch file folder and the data entered into CLASS™. The folder is submitted to Laboratory Information Services for storage in the secured central filing location.

Standards--Prior to analysis, stock standard solutions and working solutions covering the working range of the method are prepared. Procedures used in preparing the standards are recorded in standard preparation logbooks. The following information is recorded:

1. Reference standard source,
2. Lot number,
3. Date of preparation,
4. Analyst's name or initials,
5. Actual weight measured,

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6. Volumetric flask volume,
7. Calculated concentration,
8. Solvent name and lot number,
9. Dilutions, and
10. Expiration date.

Immediately after an analytical standard has been prepared, the standard is transferred to an amber glass vial, bottle, or appropriate container and properly labeled. Standards are refrigerated when not in immediate use.

12.1.1.2 GC/MS

Extraction Logbook--Once a batch has been established, the sample extraction and analysis procedure begins. An extraction logbook copy, filled out by the analyst performing the sample extraction, accompanies each lot of samples throughout analysis.

This sheet includes at least the following data:

1. Project name,
2. Extractor's initials,
3. Type of sample matrix,
4. Field group name,
5. Sample numbers,
6. Date extracted,
7. Analytical method,
8. Initial volume or wet weight of sample extracted,
9. Initial pH (water sample),
10. Extracting solvent,
11. Final volume/solvent,
12. Extract box identification,
13. Date of cleanup (if required),
14. Notes and comments affecting the extraction procedure, and

15. Surrogate/spike preparation reference number and spike volume.

After extraction is complete, the extraction logbook copies accompany the sample arrival notice to the instrumental analyst. The extracted samples are refrigerated and stored in boxes, in a central location, until the required analysis. The box number is referenced on the extraction logbook copy. Each extract vial is properly labeled and include the following information:

1. Project name,
2. Field group name,
3. Sample number,
4. Analyte group and matrix,
5. Date extracted, and
6. Extraction logbook reference number.

Spectral Data and GC/MS Computer Quantitation Report--The quantitative sample and standard data generated by the GC/MS data system and all mass spectral information are labeled and placed in the batch file folder. Manual data reduction is indicated by the flag "M" on the quantitation report.

Standards--Prior to analysis, stock standard solutions and working solutions covering the working range of the instrument are prepared. Procedures used in preparing the standards are recorded in standard preparation logbooks. The following information is recorded:

1. Reference standard source,
2. Lot number,
3. Date of preparation,
4. Analyst's name or initials,
5. Actual weight measured,
6. Volumetric flask volume,

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7. Calculated concentration,
8. Solvent name and lot number,
9. Dilutions, and
10. Expiration date.

The analytical standard is transferred immediately after preparation to a properly labeled amber glass vial, bottle, or appropriate container. Standards are refrigerated when not in immediate use.

GC/MS Instrument Logbooks--Whenever the GC/MS is used for sample analysis, the following information is recorded in an instrument logbook:

1. Instrument conditions of the gas chromatograph,
2. Instrument conditions of the mass spectrometer,
3. Analyst's initials,
4. Date of sequence,
5. Sample number or other identification,
6. Dilution factor,
7. File reference number (FRN), and
8. Method reference.

Compound Identification--Compound identification is made in terms of the full-scan mass spectrum obtained in the electron impact mode at 70 electronvolts (eV). Compound identification requires the presence of all significant major ions at the appropriate relative abundance as obtained with an authentic compound or reference spectrum from a reputable literature source. The selection of significant ions is strongly compound dependent, and because of this and other considerations, the identification of compounds entails considerable professional judgment and experience.

The most convincing evidence for compound identification is comparison of spectrum with that of an authentic compound obtained under identical operation conditions. When this is not possible due to compound availability, computer identification or library search is used and flagged as tentative identification.

Compound Quantification--The technique of extracted ion current profiles is employed for the preliminary qualitative searching and for quantification of individual compounds. Appropriate internal standards are employed to permit quantification in terms of the relative response to these internal standards. Concentration calculations and data reduction procedures are given in Section 12.1.

Spiking with Internal Standards--All samples are spiked with quantitation standards prior to the GC/MS analysis. Appropriate internal standards are selected for the remaining categories.

GC/MS Instrumental Detection Limits--The instrumental detection limit refers to the least quantity of material required to provide a total mass spectrum, of sufficient quantity, to permit compound identification. The mass spectrum contains all major ions with the appropriate relative abundance within 20 percent of either an authentic compound analyzed under identical conditions or an appropriate reference spectrum from the literature.

Data Management--Raw data such as mass spectral chromatograms, as well as calculated results, are stored on magnetic tape. Various reports present the calibration, tune, and on-column/final results. Magnetic tapes are uniquely identified, with data stored sequentially, to allow easy retrieval. Final GC/MS data results are transmitted to CLASS™ by project and sample number. The analyst processes the transmitted data and generates a batch report. The batch folder, containing the quantification report, batch

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report, copies of logbooks, and other pertinent raw data is turned into Laboratory Information Services for storage in the secured central filing location.

12.1.1.3 Trace Metals

Digestion or Sample Preparation Logbook--A copy of the digestion or sample preparation logbook, filled out by the analyst performing the sample digestion or sample preparation, accompanies each lot of samples throughout the analysis. This logbook copy will include the following data:

1. Method used (GFAA, CVAA, ICAP)
2. Analyst's initials,
3. Date sample digested,
4. Initial volume or weight,
5. Final volume,
6. Spiking solution used and standards preparation reference number,
7. Field Group,
8. Sample numbers, and
9. Notes or comments affecting the digestion procedure.

For ICAP, the ICAP computer produces a data file that is evaluated and transmitted to CLASS™. The analyst then generates a batch for review. The batch folder containing the batch report, the data file, copies of logbooks, and all other pertinent raw data are submitted to Laboratory Information Services for storage in the secured central filing location.

Laboratory Logbooks--Each instrument has its own laboratory logbook. After each analysis, the analyst records the following information in the logbook:

1. Problems encountered during the analysis,
2. Comments about the samples and/or analytical procedure,
3. Instrument used,

4. Method used (GFAA, CVAA, ICAP),
5. Date of analysis,
6. Analyst(s),
7. Element,
8. Instrument conditions,
9. Preparation logbook reference number,
10. Preparation batch reference number, and
11. Sample numbers.

Standards--Stock standard solutions are purchased from vendors. These stock solutions are certified by the vendor for purity and concentration.

Standard preparations are recorded in a logbook. The information recorded includes preparer's name, lot number, date of preparation, volumes used, calculated concentrations, and dilutions.

Volumetric dilutions are made from the stock solution to obtain working solutions. Serial dilutions are then made from the working solutions to obtain working standards to be used to generate standard curves. Working standard solutions are stored in volumetric flasks and properly labeled with the following information:

1. Preparer's name or initials,
2. Date of preparation,
3. Element(s),
4. Concentration, and
5. Expiration date (if not prepared daily).

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12.1.1.4 Inorganics

Raw data for most inorganic analyses is documented through the use of parameter logbooks. The logbooks may vary slightly in format dependent upon the type of analysis, but, at a minimum contain the following:

1. Analysis date,
2. Parameter,
3. Standard curve range and responses (where applicable),
4. Analytical batch number,
5. Instrument conditions (where applicable),
6. Method reference,
7. Sample, standard, QC sample and blank identification and responses or concentration as applicable, and
8. Analyst's initials.

Raw data for specialized instrumental analyses are documented in the following sections.

Inorganic Analysis by Autoanalyzer

After the data has been recorded in the parameter logbook, the raw data is placed in a batch file folder with copies of the notebook pages and any additional related information. These data are entered manually uploaded to CLASS™ to generate a uniquely numbered batch. The batch is reviewed for correctness and is submitted for review and finalization. When review and finalization are complete, the reviewer signs and submits the batch to Laboratory Information Services for storage in the secured central filing location.

Laboratory Logbooks--Each analytical parameter has its own laboratory logbook. During analysis, the following information is recorded:

1. Date of analysis,
2. Parameter,

4. Analytical batch number,
5. Method reference,
6. Instrument conditions,
7. Calibration standard setting and response,
8. Standard curve range, responses, and date of curve preparation,
9. Sample, standard, QC sample, and blank identification and responses or concentrations, and
10. Analyst's initials.

Inorganic Analysis by Ion Chromatography

Chromatograms--All information on the chromatograms from each analytical run is electronically recorded from the input provided during run set up. This information includes the following:

1. Analyst's initials,
2. Analytes,
3. Analysis date and time,
4. Instrument identification,
5. Integration parameters,
6. Sample, standard, and QC sample identification with concentrations and responses, and
7. Dilution factors when appropriate.

These data are manually entered into CLASS™ and an unique batch number is assigned. The data are reviewed by the analyst for correctness and submitted for review and finalization. When review and finalization are complete, the reviewer signs and submits the batch to Laboratory Information Services for storage in the secured central filing location.

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Laboratory Logbooks-The instrument has its own laboratory logbook. The following information is recorded in the logbook during the set up of the analytical run:

1. Analysis date,
2. Analyte,
4. Instrument identification and operating conditions,
5. Calibration standards and preparation dates,
6. Notes and comments as appropriate, and
7. Sample and QC sample identification numbers with dilution factors when applicable.

12.2 DATA VALIDATION

Unless otherwise specified by the client, the following procedures for review/validation of data are employed.

12.2.1 LABORATORY ACTIVITIES

Data review is performed at the bench by the analyst. The analyst reviews preliminary data entries, calculations, holding times, precision and accuracy, and calibration checks. The analyst provides an explanation and/or corrective action for any method control parameters which are outside criteria and signs the analytical batch when ready to release the data for further processing and review. This information is relayed immediately to the Department Manager, who notifies the appropriate Project Manager and Laboratory QA/QC Coordinator.

The analyst's supervisor or a designated reviewer also reviews the analytical documentation associated with the batch (such as sample preparation/digestion/extraction logbook copies, instrument logbook copies, etc.) and any explanations or corrective actions provided by the analyst. The Department Manager or designee signs and finalizes the batch after the final review.

The Project Manager checks analytical data batches that have explanations and corrective actions. The Project Manager also reviews all final data reports for inconsistencies and completeness prior to releasing the reports to the client; qualification or flagging of data and/or QC summaries are provided as appropriate.

The Laboratory QA/QC Coordinator performs quarterly audits to check that required QC procedures are being followed. This procedure entails random review of analytical batches to see that the QC designated for the analysis is being consistently performed. A record of this audit is maintained by the QA/QC staff. The Laboratory QA/QC Coordinator has the capability to initiate and follow up on corrective actions to resolve QC problems.

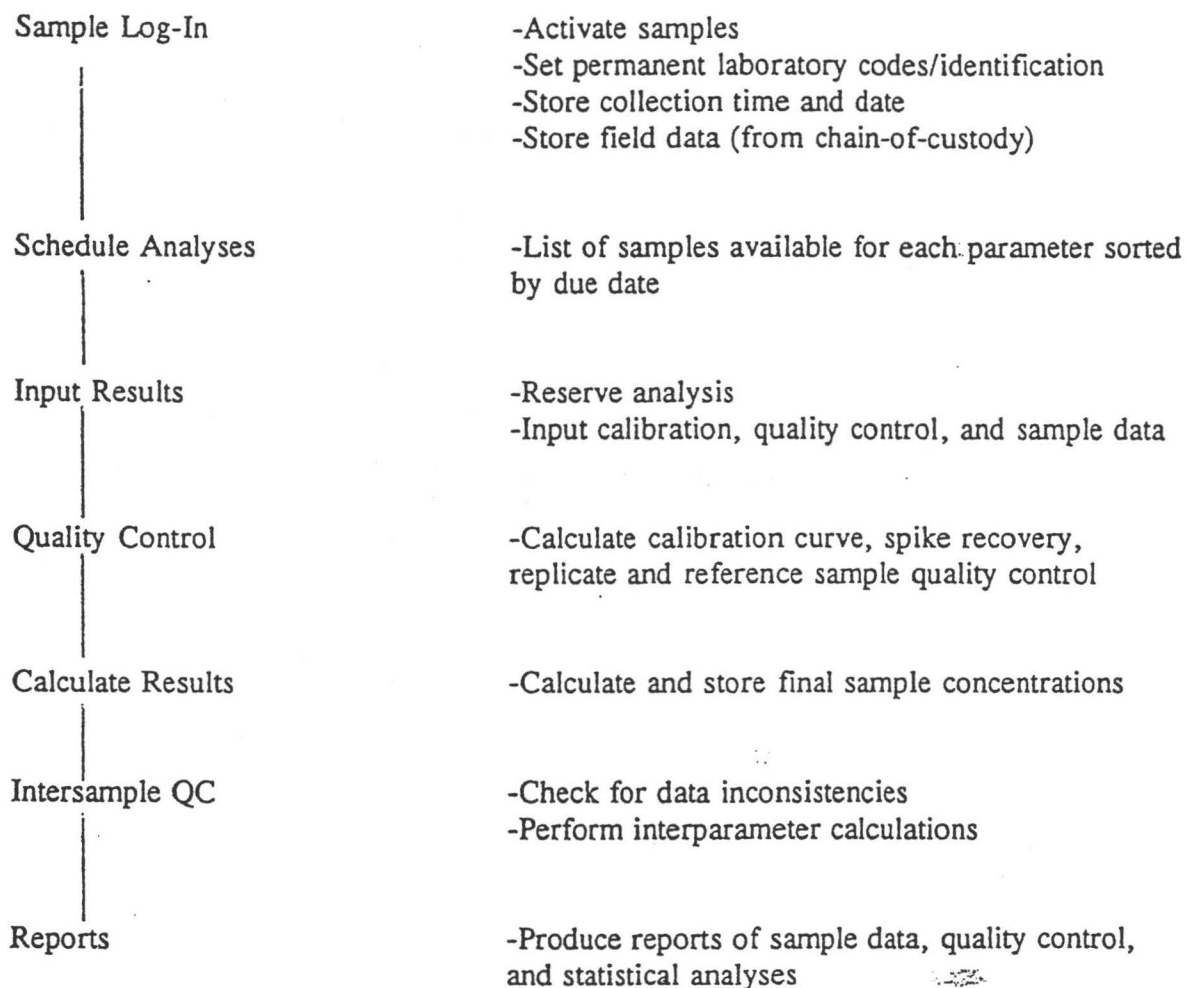
The minimum QA/QC data that should be included in the data batch are the following:

1. Sample data (matrix, date of extraction, and date of analysis),
2. Parameter, result, and test method identification,
3. Sample-specific detection limits for each parameter, and
4. Results of laboratory control data (method blanks, spikes, and replicates as required).

12.3 DATA REPORTING

Data reporting is accomplished by using CLASS™. The data flow scheme for CLASS™ is presented in Figure 12-1. All client data and pertinent field information are entered into CLASS™ directly from the chain of custody sheets. A copy of this information is given to the Project Managers for verification to ensure that all pertinent information is available and correct. CLASS™ sorts all available samples for analyses for each parameter by due date, client ID, field group, etc. Weekly reports are generated by Laboratory Information Services and sent to each analytical department to notify them of samples that are due for analysis.

Figure 12-1 Flowchart of the CLASS™ Program



Each analyst enters their analytical information into CLASS™ as a batch report. If applicable, the analysts enter standard curves (linear, quadratic, or logarithmic), method blanks, control spike data, as well as sample results into CLASS™ to create a batch. Final results are calculated according to the analytical methods specified in Section 5 of the CQAP. The analysts check all their data to ensure that all information is available and correct before signing the batch report. The analyst's Department Manager or designee then reviews the final batch report and signs it to verify that all data are accurate as reported. The batch is then finalized by the Department Manager or designee. Once a

batch is finalized, the analyst cannot change the data. Any corrections are made by the Department Manager or designee (See Section 7). The Administrative staff generates and prepares, with data from CLASS™, the final report for the client. The Project Manager reviews the final reports for inconsistencies and completeness. An example Final Report is illustrated in Figure 12-2.

12.4 DATA STORAGE

A hard copy of all batch folders, supporting documents, and project files are filed chronologically by department in the secured centralized batch storage area. The newer batch folders are also stored chronologically by department in file cabinets located in Information Services Department. The batch folders include copies of sample preparation/digestion/extraction logbooks, copies of instrument logbooks, standard preparation logbook pages, sample arrival notices, CLASS™ batch reports, and raw data. The batch folders may be checked out for review by laboratory analysts, Laboratory Coordinators, or other laboratory personnel. In addition, any personnel checking out a batch folder from Laboratory Information Services is required to sign, date, indicate the batch numbers, and department numbers on the Document Control Logbook (Figure 12-3). When the laboratory analysts, Laboratory Coordinators, or other laboratory personnel are finished reviewing the batch folders, they are returned to Laboratory Information Services and the Document Control Logbook is signed and dated. At a minimum, all project files are kept for ten years.

The original laboratory logbooks and analysts logbooks are used until they are filled and are archived by the Department Manager.

All data stored in the CLASS™ database are backed up every weekday using high-density storage media. Tapes are stored in special files and are archived in a secured air-conditioned location (CLASS™ is discussed in further detail in Section 7).



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An IEPA Contract Laboratory

TO:

REPORT DATE: 08-02-94

DATE RECEIVED: 07-25-94

PROJECT NUMBER:

ATTN:

Figure 12-2 Example Final Report

ESE SAMPLE						
SAMPLE DATE		07/22/94				
DESCRIPTION	UNITS	GRAB WATER	METHOD NO.	DATE ANALYZED	ANALYST	

METAL						

IRON	MG/L	13.2	200.7	07-27-94	ELZ	
OTHER PARAMETERS						

PH	UNITS	7.38	150.1	07-26-94	AMH	
TSS (RESIDUE, SUSP.)	MG/L	28	160.2	07-26-94	AMH	
TDS (RESIDUE DISS,180 DEG)	MG/L	597	160.1	07-26-94	AMH	
CHLORIDE	MG/L	87	4500C1B	08-01-94	KMC	

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Report Approved by:

Janel A. Woodin
Project Manager

Figure 12-3 Document Control Logbook

DOCUMENT CONTROL

[illegible]

13.0 CORRECTIVE ACTION

Corrective action is necessary whenever uncontrolled deviations from the quality assurance system occur. Quality system deviation can be detected in a number of ways, some of which include routine quality control activities, data review/verification (at all levels), performance samples, audits or other internal or external evaluations. The quality system encourages the identification and resolution of quality system anomalies at the lowest possible level, preferably by the employee responsible for performing the specific task. The effect of identified variations from the quality system range from minor to a significant quality impact and, as such, the corrective action will be based on the projected quality consequences of the identified concern.

Regardless of the source or the projected impact of the quality system deviations, the following systematic approach is recommended in developing a suitable corrective action. The emphasis of the corrective action process is to prevent the problem from recurring.

1. Define the problem.
2. Establish the root cause of the problem.
3. Determine course of action to resolve the problem and eliminate the root cause.
4. Assign responsibility for implementing the corrective action.
5. Verify that the corrective action has solved the problem and eliminated the cause.

Corrective actions in the laboratory are documented and tracked using the Data Review/Data Exception Report form and the Corrective Action Form (Figure 13-1 and Figure 13.2).

Figure 13-1 Data Review/Data Exception Report



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Data Review/Data Exception Report

SECTION 1: Completed by Analyst or Data Reviewer

Analytical Section: GC/HPLC ☐ GC/MS ☐ Inorganics ☐ Metals ☐ Other ☐ Client and Field Group: _____/_____
Method/Parameter: _____ Project Manager: _____
Matrix: _____ Date Received: ____-____-____
Analyst: _____ Samples Affected: _____
Date of Occurrence: ____-____-____ Analysis Date: ____-____-____

Problem Incurred:

QC Standard: ☐ Sample Duplicate: ☐ MS/MSD (SPM): ☐ Limited Sample Volume: ☐
Surrogate: ☐ Lab Control (SP): ☐ Blank: _____ Other: _____

Limits Exceeded:

Extractor: _____ Concentrator: _____
Control Limits Exceeded: Sample Recovery: _____ Control Limits _____ to _____ (Please list additional compounds below.)
Name of compound: _____
Comments: _____

Additional:

Matrix Interference Confirmed: ☐ Instrumental Problem: ☐ Other: ☐
Specify: _____

SECTION 2: Completed by Department Manager

Analysis Results: Accepted: ☐ Sample(s): Re-extracted/Re-prepped: ☐ Re-analyzed: ☐ Other: _____
Approved by Supervisor: _____ Date: ____-____-____
NOT Approved by Supervisor: _____ Date: ____-____-____
Clarification/Justification: _____

SECTION 3: Completed by Operations Manager

Approved by Operations Manager: _____ Date: ____-____-____
NOT Approved by Operations Manager: _____ Date: ____-____-____
Comments: _____

SECTION 4: Completed by Project Manager/Quality Assurance Manager

Problem handled in accordance with project QC guidelines: Yes: ☐ No: ☐
Data acceptable to release to client: Yes: ☐ No: ☐
Client Contacted: No: ☐ Yes: ☐ Contact: _____ Date: ____-____-____
Required Action: _____
Project Manager Approval: _____ Date: ____-____-____
Additional QA/QC Comments: No: ☐ Yes: ☐ Comments: _____
Quality Assurance Manager Approval: _____ Date: ____-____-____

SECTION 5: Completed by Analyst or Data Reviewer

White: Batch Folder Yellow: Operations Manager Pink: Project Manager Gold: QA Manager

Figure 13-2 Corrective Action Form

ESE - Peoria, IL		CORRECTIVE ACTION FORM	
Number:		Date:	
Section:		Person Contacted:	
Finding:			
Originator:	Date:	Response Due Date:	
Corrective Action Taken/Proposed to Correct Discrepancy:			
Corrective Action Taken to Prevent Recurrence (the cause of the discrepancy must also be included here):			
Corrective Action Taken By:	Date:	Date Corrective Action Will Be Taken:	
Corrective Action Evaluation:		Verification of Implementation:	
		Verified By:	Date:

13.1.3 QUALITY CONTROL CORRECTIVE ACTION

Quality control corrective action consists of corrective action following a failure to meet quality control criteria specified in this CQAP and the analytical methods. Actions taken consist of two types: those resolved within each analytical department at the time of analysis and those resolved outside the department which requires a corrective action form. Examples outlining the differences between these two types of corrective action are as follows:

WITHIN DEPARTMENT ACTION

QC Failure	Department Action
Tuning results for GC/MS fail criteria for EPA Method 624	Analyst retunes instrument before proceeding with analysis
Standard curve correlation coefficient is less than 0.995	Analyst investigates the problem and reruns curve and samples
Sample response falls outside of calibration curve	Analyst dilutes sample into the range of the curve and re-analyzes sample

OUTSIDE DEPARTMENT ACTION

QC Failure	Department Action
Holding times are exceeded	Notify Project Manager; Project Manager contacts client; Quality Assurance Manager is informed

The corrective action procedures that are taken by the Peoria Laboratory following a failure to meet QC criteria specified in this CQAP and the analytical methods, except for CLP protocol, are summarized in Tables 13-1 through 13-5.

On occasion, corrective actions are also initiated at the request of a client. The Quality Assurance Manager is responsible for approving the corrective action for the client in the same fashion as if it had been initiated by laboratory personnel.

(Rest of page left intentionally blank.)

Table 13-1. Summary of Corrective Action Procedures for Metals Analyzed by Graphite Furnace and Cold Vapor Atomic Absorption Spectroscopy

Quality Control	Acceptance Criteria	Corrective Action
Initial calibration verification standard (ICV)	$\pm 10\%$ of true value (GFAA) $\pm 20\%$ of true value (CVAA)	Rerun standard, if still out of control, recalibrate instrument.
Calibration blank (ICB)	$< \text{RL}$ (listed in Section 5)	Rerun the blank, if still out of control, reprocess and reanalyze the blank.
Calibration curve correlation coefficient	≥ 0.995	Rerun calibration standards, if still out of control, prepare new calibration standards and recalibrate the instrument or document why data are acceptable.
Calibration curve	Brackets all sample responses	Dilute and reanalyze within the calibration curve range or document why data are acceptable.
Continuing calibration verification standard (CCV)	$\pm 20\%$ of true value	Rerun standard, if still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV.
Method blank (MB)	$< \text{RL}$ (listed in Section 5)	Determine the cause of the blank problem, redigest set, if necessary, or document why data are acceptable.

Table 13-1. Summary of Corrective Action Procedures for Metals Analyzed by Graphite Furnace and Cold Vapor Atomic Absorption Spectroscopy (Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct problem, redigest and reanalyze samples, if necessary, or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why data are acceptable.

Note:RPD = relative percent difference.
RL = reporting limit

Source: ESE.

Table 13-2. Summary of Corrective Action Procedures for Metals Analyzed by Inductively Coupled Plasma Emission Spectroscopy

Quality Control	Acceptance Criteria	Corrective Action
Initial calibration verification standard (ICV)	$\pm 10\%$ of true value	Rerun standard, if still out of control, recalibrate instrument.
Calibration blank (ICB)	$< RL$ (listed in Section 5)	Rerun the blank, if still out of control, reprocess and reanalyze the blank.
Interference check standard (ICS)	$\pm 20\%$ of true value	Rerun standard, if still out of control, recalibrate instrument and reverify calibration.
Continuing calibration verification standard (CCV)	$\pm 10\%$ of true value	Rerun standard, if still out of control, recalibrate instrument and reanalyze all samples run since last acceptable CCV or document why data are acceptable.
Method blank (MB)	$< RL$ (listed in Section 5)	Determine the cause of the blank problem; redigest samples if necessary or document why data are acceptable.

Table 13-2. Summary of Corrective Action Procedures for Metals Analyzed by Inductively Coupled Plasma Emission Spectroscopy (Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct problem, redigest and reanalyze samples, if necessary, or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct problem, or document why data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why data are acceptable.

Note: RL = reporting limit.
 RPD = relative percent difference.

Source: ESE.

Table 13-3. Summary of Corrective Action Procedures for All Wet Chemistry Procedures

Quality Control	Acceptance Criteria	Corrective Action
Calibration curve correlation coefficient	≥ 0.995	Rerun calibration standards if still out of control prepare new calibration standards and recalibrate the instrument, or document why data are acceptable.
Calibration curve	Brackets all sample responses	Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable.
Calibration blank	$< \text{RL}$ (listed in Section 5)	Rerun the blank, if still out of control, reprocess and reanalyze the blank.
Continuing calibration verification standard (CCV)	$\pm 20\%$ of true value	Rerun standard, if still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV or document why data are acceptable.
Method blank (MB)	$< \text{RL}$ (listed in Section 5)	Determine the cause of the blank problem, reanalyze samples, if necessary, or document why data are acceptable.
Sample replicate (RP)*	See Section 5 for RPD control limits	Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.

Table 13-3.

Summary of Corrective Action Procedures for All Wet Chemistry Procedures
(Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct problem, reanalyze samples if necessary or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why the data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why the data are acceptable.

Note: RL = reporting limit.

RPD = replicate percent difference.

*Sample replicate is only required for miscellaneous inorganic parameters including residues, pH, specific conductivity, turbidity, dissolved oxygen, and % moisture analyses.

Source: ESE.

Table 13-4. Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography

Quality Control	Acceptance Criteria	Corrective Action
Calibration curve correlation coefficient	≥ 0.995	Rerun calibration standards, if still out of control, prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable.
Calibration curve	Brackets all sample responses	Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable.
Continuing calibration standard (CCS)	$\pm 15\%$ of standard initial response for GC EPA SW-846 and $\pm 10\%$ for GC EPA 600s $\pm 10\%$ of standard initial response for HPLC. Drinking water $\pm 20\%$	Rerun standard, if still out of control, recalibrate instrument and reanalyze samples when last CCS is acceptable, or document why data are acceptable.
Method blank (MB)	$<$ than RL for organics (listed in Section 5)	Determine and correct cause of the blank problem, reanalyze the samples, if necessary, or document why data are acceptable.
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why the data are acceptable.

Table 13-4.

Summary of Corrective Action Procedures for Organics Analyzed by Gas Chromatography and High Pressure Liquid Chromatography
(Continued, Page 2 of 2)

Quality Control	Acceptance Criteria	Corrective Action
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why the data are acceptable.
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct the problem, reanalyze samples if necessary or document why the data are acceptable.
Surrogates* (SUR)	See Section 5 for percent recovery control limits	Reanalyze samples with surrogates outside criteria or document why data are acceptable.

Note: RL = reporting limit.
GC = gas chromatography.
HPLC = high pressure liquid chromatography.
RPD = relative percent difference.

*Surrogate/surrogates will only be spiked in samples if specified by the method.

Source: ESE.

Table 13-5.

Summary of Corrective Action Procedures for Organics by Gas
Chromatography/Mass Spectrometry

Quality Control	Acceptance Criteria	Corrective Action
DFTPP or BFB instrument tuning	See Section 9 for tuning criteria	Retune instrument until within criteria.
Initial calibration standards	See Section 9 for calibration criteria	Rerun calibration standards, if still out of criteria, prepare new calibration standards and rerun standards.
One-point daily calibration	See Section 9 for calibration criteria	Rerun standard, if still out of control, rerun calibration curve, or document why data are acceptable.
Method blank (MB)	< two times the RL (listed in Section 5) for semivolatile organics	Evaluate the impact of the presence of any target analytes in the method blank, the presence of low concentrations of phthalate are acceptable. Reextract and reanalyze samples if presence of target analytes are unacceptable or document why data are acceptable.

Table 13-5.

**Summary of Corrective Action Procedures for Organics by Gas
 Chromatography/Mass Spectrometry (Continued, Page 2 of 3)**

Quality Control	Acceptance Criteria	Corrective Action
Method blank (MB)	No greater than 5 times the RL for methylene chloride, acetone, toluene, and xylene for volatile organics. All other analytes \leq RL (listed in Section 5)	Reanalyze another MB. If second MB exceeds criteria, clean and recalibrate the analytical system or document why data are acceptable.
Surrogate (SUR)	See Section 5 for percent recovery control limits	If surrogates in the MB or SP are within limits, qualify the data. Reanalyze samples with surrogates outside criteria or document why data are acceptable.
Standard matrix spike (SP)	See Section 5 for percent recovery control limits	Determine and correct the problem, reanalyze samples if necessary, or document why data are acceptable.

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Table 13-5.

Summary of Corrective Action Procedures for Organics by Gas
Chromatography/Mass Spectrometry (Continued, Page 3 of 3)

Quality Control	Acceptance Criteria	Corrective Action
Sample matrix spike (SPM)	See Section 5 for percent recovery control limits	Determine and correct the problem, or document why the data are acceptable.
Sample matrix spike duplicate	See Section 5 for RPD control limits	Determine and correct the problem, or document why the data are acceptable.

Note: RL = reporting limit.
RPD = relative percent difference.

Source: ESE.

14.0 PERFORMANCE AND SYSTEM AUDITS AND PERSONNEL TRAINING

14.1 INTRODUCTION

Two types of periodic audit procedures are used to assess and document performance of laboratory staff: system audits and performance audits. These audits form one of the bases for corrective action requirements and constitute a permanent record of the conformance of measurement systems to QA requirements.

14.2 SYSTEM AUDITS

System audits are inspections of training status, records, QC data, calibrations, and conformance to SOPs without the analysis of check samples. System audits are performed periodically by the Quality Assurance Manager.

The system audit protocol for the laboratory is summarized as follows:

1. Laboratory Operations - The Quality Assurance Manager will perform the periodic laboratory system audit using the checklist in Figures 14-1 through 14-4.

The items to be reviewed are:

- a. Parameter and/or laboratory notebooks,
- b. Instrument logbooks,
- c. Sample log-in, dispensing, and labeling for analysis,
- d. QC criteria update for spike recoveries, and
- e. Verify that deficiencies in the last audit were corrected.

In addition, the QA Manager monitors methods randomly to assure adherence to approved analytical methods.

2. Final Reports - As a normal work process, the Project Manager reviews all final reports and deliverables before they are sent to the client.

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Figure 14-1 Checklist For Coldrooms, Freezers and Sample Storage Areas

Coldrooms, Freezers and Sample Storage

Department: _____

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available for receipt, storage, and tracking of samples?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are the Sample Tracking forms properly filled out?			
6. Is the Sample Location report updated on a regular basis and placed next to the door of each storage area?			
7. Are all storage areas secured at all times?			
8. Are the temperature logs for the coldrooms and freezers filled out completely and corrections made properly? Are appropriate corrective actions taken for all out-of-control readings?			
9. Is a condensed SOP for check-in/check-out log filled out completely?			
10. Is the Sample Check-In/Check-Out log filled out completely?			
11. Is proper documentation available for tracking the disposal of samples?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm. #, logbook #, Page #, instrument #, etc.)

Figure 14-2 Checklists For Sample Receiving and Hood Maintenance

Sample Receiving

Department: _____

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available for receipt, log-in and transfer of samples to storage areas?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials?			
5. Is the Sample Custodian filling out all required information on the chain of custody (COC) form (cooler temp., seals intact? etc.)?			
6. Are the Sample Chest Custody Forms filled out completely?			
7. Is the Sample Custodian completely filling out the Cold Room Sample Arrival logbook?			
8. Is the Sample Custodian auditing 10% of all samples (except VOA samples) to verify that samples are properly preserved? Is documentation available?			
9. Are samples labelled properly?			
Additional Comments:			

Hood Maintenance

Department: _____

ITEM	YES	NO*	Comments
1. Have fume hoods been calibrated within the last year? Are they labelled as to when last tested?			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm#, logbook #, page #, instrument #, etc.)

Figure 14-3 Checklist For Sample Kit Prep Area

Sample Kit Prep Area

Department: _____

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are all preservatives labelled properly?			
6. Is the sample Kit Prep & Shipping Request Form filled out completely?			
7. For coolers picked up[by field personnel, is the appropriate information documented in the Kit Pick-up log? Is the Kit Pick-up log signed by both kit prep and field personnel?			
8. For coolers shipped to the field, is the appropriate information documented in the Shipping receipt (ice chest check out) log?			
9. Is a copy of the Shipping Receipt (ice chest check out) form attached to the Kit Prep & Shipping Request form?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm#, logbook #, Page #, instrument #, etc.)

Figure 14-4 .
Checklist For Laboratory Area Responsibilities and Glassware Washing Procedures

Laboratory Area Responsibilities

Department: _____

ITEM	YES	NO*	COMMENTS
1. Have fume hoods been calibrated within the last year? Are they labelled as to when last tested?			
2. Are refrigerator/freezer temperature logs filled out completely and corrections made properly? Are temperatures taken daily, except weekend days? Are appropriate corrective actions taken for any out-of-control readings?			
3. Are the balance calibration logs filled out completely and corrections made properly? Are balances calibrated daily, except weekend days, for analytical balances and weekly for top loading balances? Are appropriate corrective actions taken for any out-of-control readings?			
4. Is the balance manufacturer's maintenance done annually?			
5. Are documentation errors for these logbooks corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
Additional Comments:			

Glassware Washing Procedures

Department: _____

Item	Yes	No*	Comments
1. Is the work area clean and organized?			
2. Are SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Is clean glassware stored so as to avoid contamination?			
6. Are the Glassware Washing Request Forms filled out completely and signed and dated?			
7. Are properly labelled waste containers available?			
8. Is the deionized water system checked regularly to verify that it meets requirements?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm#, logbook #, page #, instrument #, etc.)

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Figure 14-5 Checklist For Sample Preparation Areas

Sample Preparation Areas

Department: _____

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available for receipt, storage and tracking of samples?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are samples and standards stored separately to avoid contamination?			
6. Are spike solutions, surrogate solutions, (Org. only) and reagents labelled clearly and appropriately (including plastic squeeze bottles)?			
7. Are there expired standards/reagents in the laboratory? Are they clearly labelled as "expired" or "for qualitative use only"?			
8. Is glassware stored so as to avoid contamination?			
9. Do all log books have control numbers?			
10. Are sample preparation logs completely filled out, including preparer and reviewer signatures?			
11. Are automatic pipettes and syringes calibrated each day of use? (Inorganics Division only) Are all water bath thermometers in use calibrated against a NIST thermometer? (Organic Division) Are the calibrations documented in the appropriate logbooks?			
12. Are instrument run logs made properly (e.g., microwave, GPC)?			
13. Are instrument maintenance logs filled out completely and corrections made properly?			
14. Are extracts (sample vials) labelled properly?			
15. Are sample extract/digest chain of custody logs filled out completely and corrections made properly?			
16. Are properly labeled waste containers available?			
Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-6 Checklist For Sample Analysis Area

Sample Analysis Areas

Department: _____

ITEM	YES	NO*	COMMENTS
1. Is the work area clean and organized?			
2. Are SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are samples and standards stored separately to avoid contamination?			
6. Are spike solutions, surrogate solutions (Org. only), calibration standards and reagents labelled clearly and appropriately (including plastic squeeze bottles)?			
7. Is glassware stored so as to avoid contamination?			
8. Do all logbooks have control numbers?			
9. Are standard and reagent prep. logbooks filled out completely and corrections made properly? Are lot numbers of neat standards recorded?			
10. Are instrument calibration checks performed prior to analysis? (Mandatory for Radiochemistry, only)			
11. Are instrument run logs filled out completely and corrections made properly?			
12. Are instrument maintenance logs filled out completely and corrections made properly.			
13. Are samples (analysis vials) labelled properly?			
14. Are sample chain-of-custody (COC) logs (VOA samples) or sample extract/digest COC logs filled out completely and corrections made properly?			
15. Are properly labeled waste containers available?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

Figure 14-7

Checklist For Information Services

Information Services

Department: _____

ITEM	YES	NO*	COMMENTS
1. Is the work area organized?			
2. Are appropriate SOPs available?			
3. Are there findings in this department from last quarter's lab audit? If yes, list below (or attach a separate sheet) and verify that they have been corrected.			
4. Are documentation errors corrected properly (one line drawn through error, date, error code/explanation, and initials)?			
5. Are the Chain-of Custody Forms properly filed and readily accessible?			
6. Are the filing cabinets where data are stored kept locked?			
7. Are batch folders readily accessible?			
8. Is the Document Control Logbook filled out completely?			
9. Are the appropriate approval forms and signatures maintained for changes to finalized data batches or CLASS™ STORET files?			
Additional Comments:			

*For all "No" answers, include all information necessary to trace audit finding (e.g., Rm.#, logbook #, page #, instrument #, etc.)

The Peoria Laboratory is audited periodically by external sources, such as state and federal agencies. These formal external audits are conducted to verify compliance with rules, regulations, or criteria for certification. The Peoria Laboratory is externally audited regularly by the following agencies:

1. State of Illinois Environmental Protection Agency,
2. State of New Jersey Department of Environmental Protection and Energy,
3. State of California Department of Health Services,
4. State of New Hampshire Department of Environmental Services,
5. State of Wisconsin Department of Natural Resources,
6. State of Florida Department of Health and Rehabilitative Services,
7. State of North Carolina Department of Environment, Health, and Natural Resources, and
8. United States Army Corps of Engineers.

ESE submits to periodic external audits after notification and scheduling by the QA Manager and the Laboratory Director.

14.3 PERFORMANCE AUDITS

Performance audits are inspections of the on-going quality program in the laboratory focusing on the evaluation of the accuracy of all laboratory data.

The results of interlaboratory studies are evaluated by the QA Manager as part of the performance audits. This type of evaluation is performed at least quarterly. ESE participates in the following proficiency programs:

1. USEPA Water Pollution and Water Supply proficiency programs,
2. USEPA National Pollutant Discharge Elimination System (NPDES) DMR-QA proficiency program,
3. American Industrial Hygiene Association (AIHA), Environmental Lead Proficiency Analytical Testing (ELPAT) program,

4. State of Wisconsin, State Laboratory of Hygiene,
5. U.S. Army Corps of Engineers, and
6. Analytical Standards, Inc., Environmental Performance Audit (EPA)[™] program.

Besides participation in several proficiency programs, the ESE Peoria Laboratory is currently certified by numerous state and regulatory agencies which require verification of laboratory's proficiency on an annual basis. The following licenses, accreditations, certifications and validations are held by the Peoria Laboratory:

1. State of California Department of Health Services,
2. State of Connecticut Department of Health Services,
3. State of Florida Department of Health and Rehabilitative Services,
4. State of Illinois Environmental Protection Agency,
5. State of Illinois Contract Laboratory Program,
6. State of Iowa Department of Natural Resources,
7. State of Kansas Department of Health and Environment,
8. State of Kentucky Department For Environmental Protection,
9. State of New Hampshire Department of Environmental Services,
10. State of New Jersey Department of Environmental Protection and Energy,
11. State of North Carolina Department of Health, Environment, and Natural Resources,
12. State of Oklahoma Water Resources Board,
13. State of Washington Department of Ecology,
14. State of Wisconsin Department of Natural Resources, and
15. United States Army Corps of Engineers.

In addition to reviewing performance evaluation program results, the QA Manager performs a data review on a basis of at least ten percent of all batches generated. On a

14.4 PERSONNEL TRAINING

The Peoria Laboratory personnel are trained in health and safety, QA/QC procedures, analytical methods, and the laboratory data management system. New personnel are trained prior to performing any actual laboratory work. Laboratory personnel are also required to attend the health and safety and laboratory QA/QC procedures refresher courses offered yearly. Training that each laboratory personnel receives is documented in the personnel's training records.

15.0 QUALITY ASSURANCE REPORTS

Project Quality Assurance reports are either internal or external in nature. Upon request, a Project QA report is written upon completion of the project or immediately upon the discovery of a problem requiring corrective action. The Inorganic and Organic Operations Manager is responsible for compiling the QA information provided by the Department Managers and submitting the complete report to the client/agency. Activities and actions to be reported will include:

1. An assessment of the project's status in relation to the progress of proposed time table;
2. Results of ongoing performance and system audits (Results of other performance and system audits are reported to management quarterly by the Laboratory QA/QC Coordinator);
3. Assessment of measurement data accuracy, precision, and method detection limits; and
4. Data quality review and significant QA problems with proposed corrective action procedures.

The Department Managers, Project Managers, and Laboratory QA/QC Coordinator are informed of the contents of the final Project QA report by the Inorganic and/or Organic Operations Manager through review of the final report.

16.0 PERSONNEL SUMMARY AND RESUMES

Table 16-1 lists the titles and positions of all laboratory personnel currently employed at the ESE Peoria Laboratory.

ESE PEORIA ANALYTICAL LABORATORY

Personnel Summary

TITLE	NAME	DEGREE/YEAR BACKGROUND	YEARS EXPERIENCE
Laboratory Director	Kim D. Johnson	B.S., 1989, Business Management, Laboratory Director	16
Customer Services Manager	Kim D. Johnson	B.S., 1989, Business Management, Laboratory Director	16
Laboratory Project Manager	Vickie M. Wynkoop	B.S., 1978, Biology, Project Management	12
Laboratory Project Manager	Karri L. Derr	B.S., 1988, Animal Science, Project Management	8
Laboratory QA Manager	Michael A. Travis	B.A., 1976, Chemistry, Mass Spectrometry; QA/QC	12
Department Manager-- Laboratory Information Services/Sample Receiving	Dean J. Huhmann	B.S., 1986, Management Information Systems LIMS Management	8
Staff Lab Scientist	Dave Hampson	B.A., 1969, Biology, B.S., 1978, Pharmacology Sample Receiving	4
Department Manager--GC/MS	Glen A. Coder	B.A., 1990, Communication Arts and Sciences, Mass Spectroscopy	5
Staff Lab Scientist	Doug A. Hafley	B.S., 1990, Chemistry, Mass Spectrometry	9
Senior Staff Lab Scientist	Steve Marsh	B.S., 1989, Biology, Mass Spectrometry	7
Staff Lab Scientist	Todd J. Peterson	B.S., 1991, Chemistry, Chromatography, Organic Extractions	4
Department Manager-- GC/HPLC	Troy A. Avery	B.S., 1994, Chemistry, Chromatography; Mass Spectroscopy	2
Senior Staff Lab Scientist	Sandra K. Boucher	B.S., 1974, Biological Science, Mas Spectrometry; Chromatography	7

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TITLE	NAME	DEGREE/YEAR BACKGROUND	YEARS EXPERIENCE
Staff Lab Scientist	Judy A. Zosky	High School, Chromatography	4
Extraction Group Leader	Jeff Olson	B.S., 1988, Chemistry, Organic Extractions, Chromatography	8
Staff Lab Scientist	Wei Q. Zhong	B.S., 19845, Biology, Organic Extraction	2
Lab Technician I	Bruce Ebb	A.S., 1996, Med. Lab. Tech., Organic Extraction	<1
Senior Staff Lab Scientist	Gregory R. St. Aubin	B.S., 1988, Agriculture/ Agronomy, Spectroscopy; Inorganic Chemistry	6
Staff Lab Scientist	Deborah A. Blahnik	LPN, 1973 Inorganic Sample Preparation	9
Senior Staff Lab Scientist	Ellen L. Smith	B.S., 1988, Biology, Spectroscopy; Inorganic Chemistry	6
Administrative Assistant-- Financial	Sandra D. Frye	High School, Administration	29
Administrative Assistant	Joan M. VanLoo	High School, Administration	16
Administrative Assistant	Amy K. Smith	High School, Administration	6

APPENDIX A LABORATORY FACILITIES

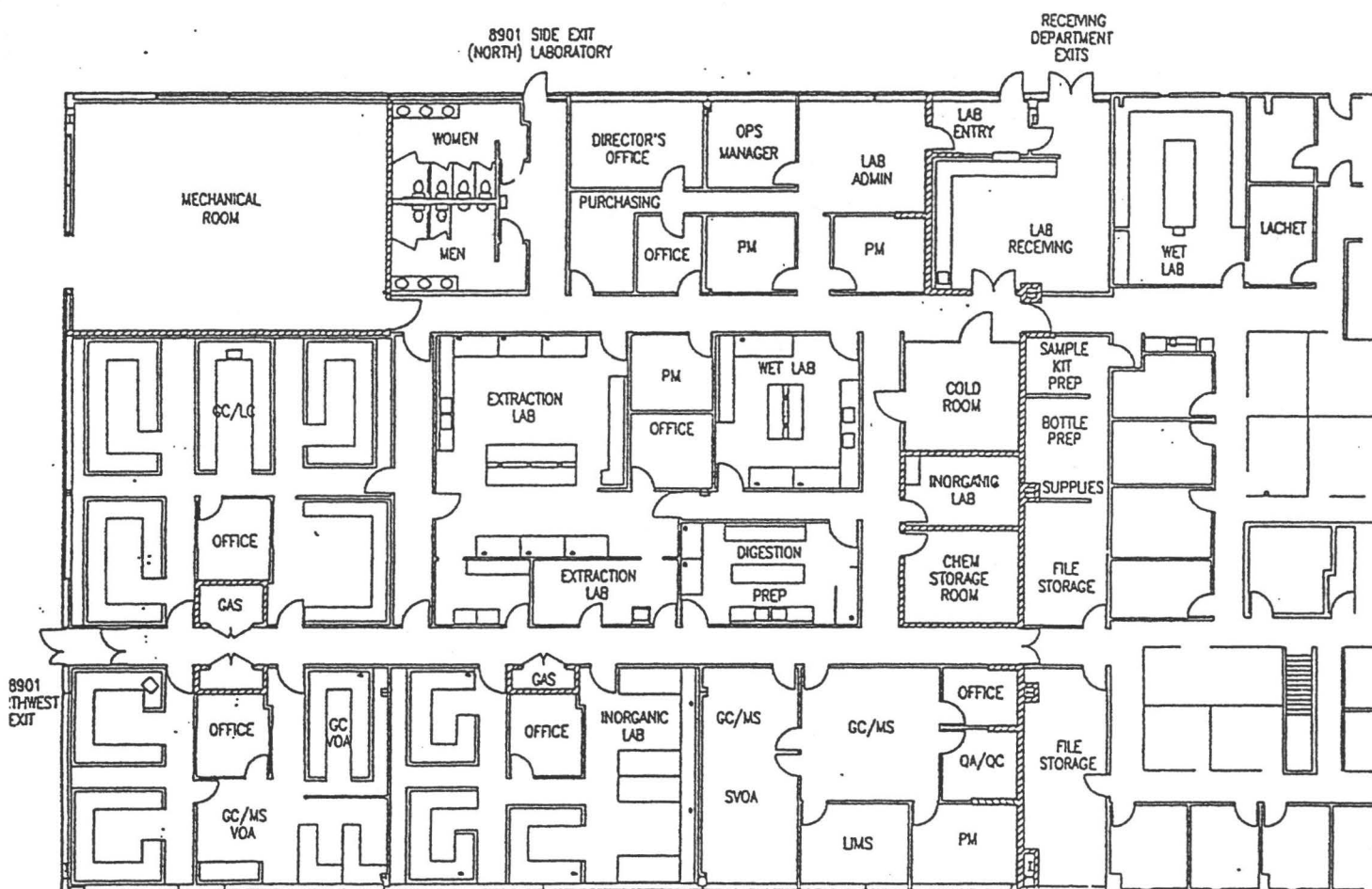
The Environmental Science & Engineering, Inc. facility in Peoria, Illinois has over 17,000 square feet of laboratory, office, computer, and storage space. The facilities have been designed with efficient operations and safety in mind.

The laboratory has dedicated areas for organic extraction, inorganic preparation, metals digestion, GC/MS, GC and HPLC analysis, ICP and AA analysis, classical water quality analysis, toxic chemicals handling, and additional support areas housing ovens, analytical balances, glassware washing, kit preparation, chemicals storage, and waste storage. The GC/MS and GC laboratories have been divided to provide separate rooms for the analysis of volatile organics and semivolatile organics in order to minimize cross-contamination. Benchtops throughout the laboratory are corrosion-resistant, all walls and floors are non-absorbent, and good housekeeping practices are stressed. The laboratory section managers are responsible for ensuring the order and cleanliness of their individual areas. Preventative maintenance, cleaning, and repairs are conducted in a timely manner to assure performance to specification.

The laboratory is supplied with demineralized water for glassware washing and other functions. Supplies of organic-free water is maintained at all times for use in trace organic analysis.

An electronic security system is used to control access to the facility. The primary source of entry is into the main reception area. Admittance to the facility is permitted by magnetic key card or by the receptionist. Other points of entry, such as the sample receipt area and the fire exits, are kept locked or under constant surveillance. Computer and word-processing operations, which provide most of the data handling and report generation support, are in secure areas which are locked when not occupied. The LIMS computer is maintained in its own temperature-controlled, voltage-regulated room. The LIMS software is password protected.

The laboratory facility is equipped throughout with a full range of safety equipment including fume hoods, eye washes, emergency showers, emergency lights, fire extinguishers, spill clean-up kits, smoke alarms, warning signs, lighted exit signs, safety glasses, and fire blankets. The laboratory also has a documented Chemical Hygiene Plan in operation which provides for training, information, and procedures to maintain analyst safety.



Peoria, Illinois Laboratory Floor Plan